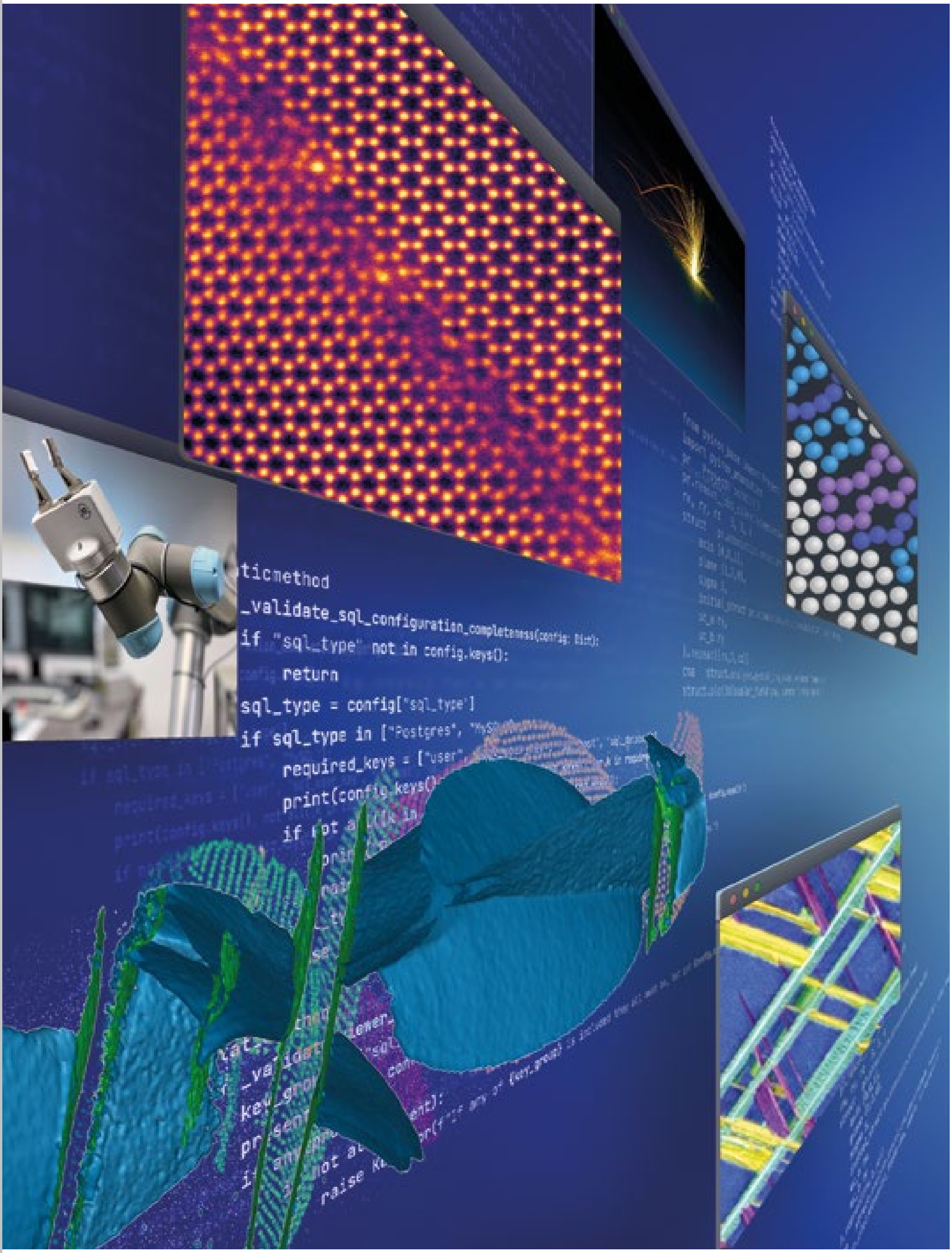
Scientific Report
2019 – 2021



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    required_keys = ["user"
    print(config.keys()
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MAX-PLANCK-INSTITUT
FÜR EISENFORSCHUNG GmbH

Max-Planck-Institut für Eisenforschung GmbH

Scientific Report 2019 – 2021

December 2021

Max-Planck-Institut für Eisenforschung GmbH
Max-Planck-Str. 1
40237 Düsseldorf
Germany

Front cover

At the MPIE, computational and experimental techniques go hand in hand to tackle the challenges of materials science and engineering. The cover shows an artist's impression of automated workflows on our integrated development platform pyiron, that can combine e.g., machine learning approaches with high-end experiments like atom probe tomography, transmission electron and robotic microscopy.

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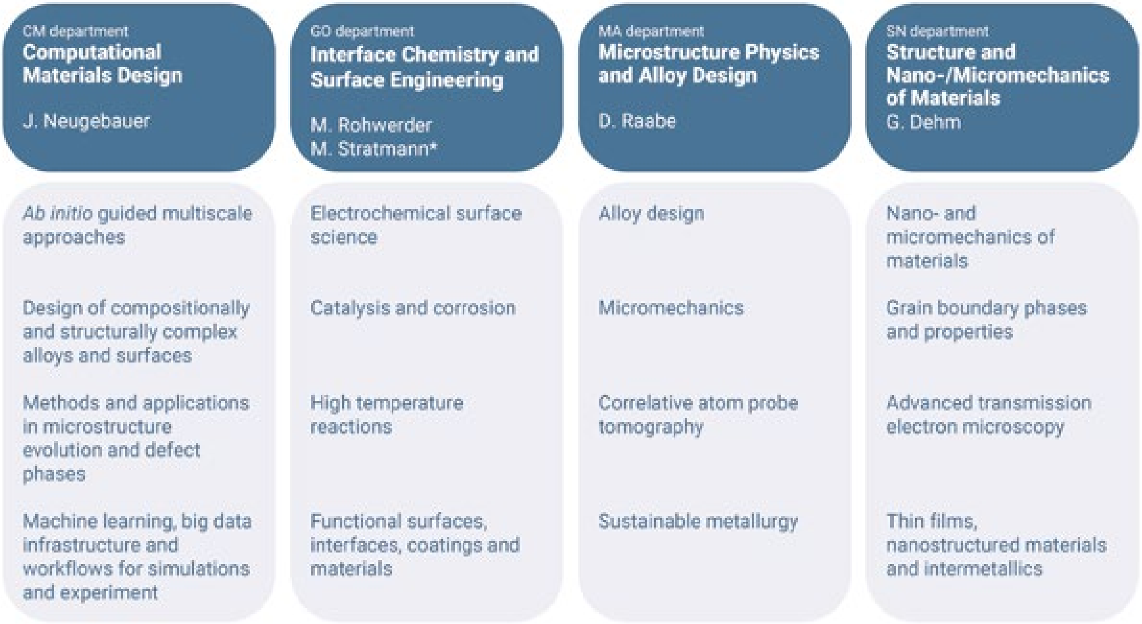
Preface

This report presents the scientific activities and achievements of the Max-Planck-Institut für Eisenforschung GmbH (MPIE) between 2019 and 2021.

The institute has four departments: Computational Materials Design (Prof. J. Neugebauer), Interface Chemistry and Surface Engineering (Prof. M. Stratmann (on leave, provisional head: Prof. J. Neugebauer)), Microstructure Physics and Alloy Design (Prof. D. Raabe) and Structure and Nano-/Micromechanics of Materials (Prof. G. Dehm). The MPIE also hosts an Independent Max Planck Research Group on Nanoanalytics and Interfaces (Prof. C. Scheu). Each department consists of several research groups. The MPIE has a number of interdepartmental and partner groups where topics of interdisciplinary content are jointly pursued. Service groups support the scientific departments. These include the synthesis, processing and testing of materials, chemical analysis, metallography, a mechanical workshop, facilities to design and build scientific equipment, an electronic workshop, a library, a computer network centre and a research coordination office.

The MPIE's mission is to understand and design materials for structural and functional applications down to atomic and electronic scales, fully including the impact of real environments. Using latest analysis methods and theory, many of them developed in-house, we start to resolve and quantify material complexity in terms of its rich nano-cosmos, manifested by hierarchical and entangled atomic-scale structural and electrochemical building units and defects. Accessing this complexity at a fundamental level is highly challenging and requires the constant development of experimental and theoretical techniques as most of the constituting nano-features are buried in the bulk, change over time (4D materials) and experience entanglements with correlations that range from a few atomic distances to several micrometres. The structural complexity of advanced materials requires also huge efforts in probing how such materials respond to external or internal chemical, physical, bio-medical and mechanical stimuli.

This mission is nowadays more essential than ever: global society enters from the age of industry into the age of circular economy, sustainability and digitalization. Advanced materials have a fundamental impact on all these fields. They can solve many of the challenges by enabling products with new features and properties. Their synthesis and manufacturing can be revolutionized by knowledge-driven workflows that integrate theory, data-driven rapid maturation and robotic production. Materials also stand for some of the most pressing burdens of our generation: Since their production causes about 50 % of all industrial CO_2 emissions and 10 % of the global energy consumption, research in this field is a matter of highest urgency.



CM department **Computational Materials Design** J. Neugebauer	GO department **Interface Chemistry and Surface Engineering** M. Rohwerder M. Stratmann*	MA department **Microstructure Physics and Alloy Design** D. Raabe	SN department **Structure and Nano-/Micromechanics of Materials** G. Dehm
Ab initio guided multiscale approaches	Electrochemical surface science	Alloy design	Nano- and micromechanics of materials
Design of compositionally and structurally complex alloys and surfaces	Catalysis and corrosion	Micromechanics	Grain boundary phases and properties
Methods and applications in microstructure evolution and defect phases	High temperature reactions	Correlative atom probe tomography	Advanced transmission electron microscopy
Machine learning, big data infrastructure and workflows for simulations and experiment	Functional surfaces, interfaces, coatings and materials	Sustainable metallurgy	Thin films, nanostructured materials and intermetallics

*M. Stratmann is on leave for the time of his presidency of the Max Planck Society. J. Neugebauer is the provisional head of the department.

Fig. 1: *Scientific scopes of the departments.*

At the MPIE we address and solve many of these challenges with novel materials, functions and processes, using interdisciplinary approaches, involving experimentalists and theoreticians from different groups and departments. The often multi-disciplinary research tasks have also motivated us to found dedicated interdepartmental research groups, to foster collaboration between the different fields. Some grand research challenges that we pursue are:

- How can materials endure hydrogen exposure; what are the links between hydrogen embrittlement, alloy chemistry and microstructure? How can hydrogen ingress be prevented or tolerated?
- How can mutually exclusive properties be reconciled in a single material system?
- What is the interplay between structure, chemistry, thermodynamics and kinetics at buried lattice defects?
- How can materials heal themselves, be more adaptive to changing environments, alter their properties on demand and maintain or even improve their response under harsh system conditions?
- How can theory- and data-driven material design methods in combination with synthesis robots and automated experimental characterization enable autonomous materials discovery, synthesis and manufacturing?
- How can basic materials science help to make the entire materials system ecologically more responsible and more sustainable, serve a circular economy and break the conventional end-of life view with practically endless recycling by proper design of materials and processes?

Some recent developments are worth to be mentioned: as of Jan.1st, 2022 the MPIE has finalized its transition into 100% ownership by the Max Planck Society, while remaining its legal entity as a GmbH until administrative processes are aligned during the next years. During the past 3 years, MPIE has used the transition phase to sharpen its research profile by adding topics related to fuels cells, hydrogen, magnetism, thermoelectrics, sustainability and machine learning to its portfolio, flanked by (bi-)weekly workshops and joint seminars, including extramural partner groups.

The MPIE also participates in several large network programs amongst others 6 collaborative research centers (SFBs) funded by the German Research Foundation (DFG) and it hosts 4 ERC grants in the reporting period (in total 7 ERC Grantees since 2012). The MPIE won in 2017 and 2020 highest ranks in the Humboldt ranking among all non-university research organizations in Germany (rank 1 in engineering, rank 3 over all sciences) with 33 awarded scholarships between 2019 and 2021.

In 2020, Dr. Baptiste Gault received the highest German research award - the Gottfried Wilhelm Leibniz Award 2020 by the German Research Foundation. Over the past 3 years more than 20 members of the MPIE gained professorships from prestigious universities around the globe, underlining the institute’s ambition to propel careers. And the MPIE heads the International Max Planck Research School - IMPRS SurMat.

The institute evolved not only scientifically, also work & life at the institute opened new areas: The MPIE now has a Team Green which is committed to create a sustainable and ecological working environment. The health management revealed that the overall working conditions are above average and so is also the overall working satisfaction. Our MPIE gender equality plan was awarded “silver” by the Max Planck Society.

This report is structured into four parts:

- *Part I* contains information on recent scientific developments, new scientific groups, large network activities, new scientific laboratories, and on the work and life at the MPIE.
- *Parts II* and *III* cover the research activities of the institute. Part II provides a description of the scientific activities in the departments and Part III contains selected highlights, which summarize major recent scientific achievements in several topical areas of common interest.
- *Part IV* summarizes statistically relevant information about the institute.

The Directors of the MPIE

Düsseldorf, December 2021

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Part I.

The institute

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Long-term oriented method development

Large-scale and networking projects

Work, family and campus life

Scientific organization

Board of Executives

Prof. G. Dehm, Prof. J. Neugebauer, Prof. D. Raabe (CEO), Dr. K. de Weldige

CM department
Computational Materials Design
Prof. J. Neugebauer

Computational Phase Studies
Dr. T. Hickel

Funded by Netherlands Organisation for Scientific Research
Complex Concentrated Alloys
Dr. F. Körmann

Computer Center
Dr. C. Freysoldt

Co-funded by EU and BMBF
Growth Modelling
Dr. L. Lymperakis

Defect Chemistry and Spectroscopy
Dr. C. Freysoldt

Electrochemistry and Corrosion
Dr. M. Todorova

Microstructure and Mechanics
Prof. E. Bitzek

Thermodynamics and Kinetics of Defects
Dr. L. Huber

GO department
Interface Chemistry and Surface Engineering
Prof. M. Stratmann*

Atomistic Modelling
Dr. S. Wippermann

RUB-MPIE Junior Research Group
Spectroscopy at Electrochemical Interfaces
Dr. M. Rabe

Corrosion
Dr. M. Rohwerder

Guest Group
Dynamic Electrocatalytic Interfaces
Dr. O. Kasian

Interdepartmental & Partner Groups

MPG Group
De Magnete Designing Magnetism
Prof. O. Gutfleisch

MPG Group
High Temperature Materials
Prof. G. Eggeler

MPG Group
Thin Film Materials Design
Prof. J. Schneider

Physical Metallurgy of Sustainable Alloys
Dr. Y. Ma
Dr. I. Souza

Max Planck Partner Group
Combinatorial Design of Permanent Magnets
Prof. K. G. Pradeep

Max Planck Partner Group
Degradation of High Temperature Materials
Prof. S. K. Makineni

Max Planck Partner Group
Functional Oxide Structures
Prof. N. J. Balila

Max Planck Partner Group
High Performance Materials for Harsh Energy Applications
Dr. J. Zavasnik

Scientific Service Groups

Hydrogen Laboratory
- Electrochemical and gas-phase hydrogen charging
- Thermal desorption spectroscopy

Dr. M. Rohwerder

Materials Testing
- Mechanical properties
- Physical properties
- Varying environments

Dr. D. Ponge

Metallography
- Light microscopy
- Metallographic sample preparation
- Scanning electron microscopy

Dr. S. Zaefferer

Synthesis and Processing
- Alloy production
- Annealing and welding
- Thermomechanical treatments

Dr. D. Ponge

Administration & Non-Scientific Service Groups
Dr. K. de Weldige

Accounting and Third Party Funds
M. Bosch

Human Resources
J. Heisel

Procurement
U. Theuerkauf

Electronics
I. Gonzalez

Boards & Representatives

Apprentice Representative
S. Becker

Disability Representative
V. Kree

Gender Equality Officer
S. Degner

Scientific Advisory Board
Coordinator: A. Schuck

*Prof. M. Stratmann is on leave for the time of his presidency of the Max Planck Society. Provisional head of the department is Prof. J. Neugebauer.

MA department
Microstructure Physics and Alloy Design
Prof. D. Raabe

Atom Probe Tomography
Dr. B. Gault

Funded by German Research Foundation
High-Entropy Alloys
Prof. Z. Li
(guest group leader)

Computational Sustainable Metallurgy
Dr. J. R. Mianroodi

Funded by German Research Foundation
High Performance Alloys for Extreme Environments
Prof. P. Kontis
(guest group leader)

Mechanism-based Alloy Design
Dr. D. Ponge

ERC Consolidator Grant
Hydrogen in Energy Materials
Dr. B. Gault

Microscopy and Diffraction
Dr. S. Zaefferer

Funded by German Research Foundation
Integrated Computational Materials Engineering
Prof. M. Diehl
(guest group leader)

Theory and Simulation
Dr. F. Roters

Funded by BMBF
Materials Science of Mechanical Contacts
Dr. M. Herbig

SN department
Structure and Nano-/ Micromechanics of Materials
Prof. G. Dehm

Advanced Transmission Electron Microscopy
Dr. C. Liebscher

Funded by ERC Advanced Grant
Atomistic Modelling of Material Interfaces
Dr. T. Brink

Intermetallic Materials
Dr. M. Palm
Dr. F. Stein

Funded by ERC Advanced Grant
Hydrogen Mechanics and Interfaces
Dr. M. J. Duarte Correa

Nanomechanical Instrumentation and Extreme Nanomechanics
Dr. R. Ramachandramoorthy

Nano-/ Micromechanics of Materials
Dr. J. P. Best

Thin Films and Nanostructured Materials
Dr. J. P. Best

Independent research groups, members and schools

Independent Max Planck Research Group
Nanoanalytics and Interfaces
Prof. C. Scheu

Max Planck Fellow Group
Electrochemistry and Nanoscale Materials
Prof. K. Tschulik

Int. Max Planck Research School
Interface Controlled Materials for Energy Conversion
E. Gattermann
Dr. S. Wippermann

External Members
Prof. M. Hillert
Prof. R. Kirchheim

Hydrogen Embrittlement in High-Performance Alloys
Dr. B. Sun

Materials Science of Additive Manufacturing
Prof. H. Springer
Dr. R. Ramachandramoorthy

Max Planck Partner Group
Stress and Defects Driven Phase Transformations
Prof. R. S. Meka

Partner Group with RWTH Aachen University
Sustainable Materials Science and Technology
Prof. H. Springer

X-Ray Diffraction

- XRD phase analysis
- Pole figure and residual stress measurements
- Advanced XRD techniques

B. Breitbach

IT Services
R. McCormack

Library
P. Siegmund

Research Coordination Office
Dr. K. Hübel

International Office	Public Relations	Research Funding	Int. Max Planck Research School
S. Zwaka	Y. Ahmed Salem	Dr. K. Hübel	E. Gattermann

Technical Services incl. Mechanical Workshop
M. Winkler

Supervisory Board
Coordinator: A. Schuck

Work Council
K. Angenendt

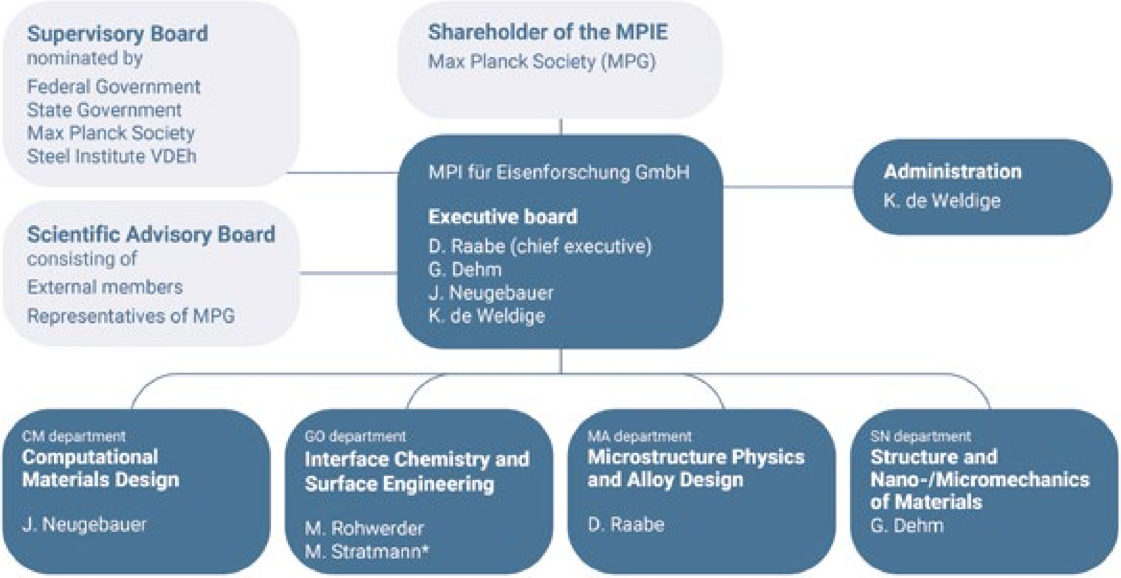
Management of the institute

Today the Max-Planck-Institut für Eisenforschung GmbH (MPIE) is fully funded by the Max Planck Society. Until 2021 it was a joint venture between the Max Planck Society and the Steel Institute VDEh.

The institute was founded in 1917 by the Verein Deutscher Eisenhüttenleute (VDEh) and incorporated into the Kaiser Wilhelm Society, the predecessor of the Max Planck Society. The MPIE was first located in Aachen and was associated with the Technical University of Aachen. Later, in 1934/35, the institute moved to its present location on a site donated by the city of Düsseldorf.

In 1946, the institute's heavily damaged buildings were reconstructed, work resumed and the MPIE was integrated into the newly formed Max Planck Society in 1948. It rapidly expanded and new laboratory buildings were built in the early 1960s. Following the appointment of H.J. Engell as director in 1971, a complete reorganization of the institute was carried out. Since then it has operated on the legal basis of a limited liability company (GmbH).

Until 2002, the institute was headed by a chief executive director (1971-1990: H.J. Engell, 1990-2002: P. Neumann) and an associated administrative director. Since June 2002, all scientific members and the administrative head of the MPIE form an executive board of directors. The position of a managing director is filled, in rotation, by one of the board members. The supervisory board reflects and debates with the MPIE management and the Max Planck Society on questions regarding the strategy and research mission of the institute. It comprises representatives from academia, industry, research and politics. Moreover, the institute's performance is evaluated every three years through a scientific advisory board. This board consists of prominent scientists and industry representatives who assist the institute in balancing fundamental research and technological relevance.



*M. Stratmann is on leave for the time of his presidency of the Max Planck Society. J. Neugebauer is the provisional head of the department.

Independent Max Planck research group: Nanoanalytics and Interfaces (NG)

Group head: C. Scheu

Research mission and scientific concepts

One central research mission of the Nanoanalytics and Interfaces (NG) group is to design and optimize materials for the whole value chain of the hydrogen technology. This starts with (photo)electro-catalytic active materials, which are used to generate green hydrogen either by light driven water splitting or with electrolysis powered by renewables [1-24]. This green hydrogen can be used in polymer-based fuel cells and the constituting material systems for these devices are also investigated [25-31]. The only by-products from fuel cell operation are water and heat. Ideally, the waste heat can be converted to electricity by thermo- electric devices, and activities in the NG group are devoted therefore to this material class [11, 32-37]. Further research fields are new materials for batteries, hydrogen storage, and energy-efficient light-emitting devices [1, 38-47].

To design and optimize renewable energy materials their microstructure and defects have to be unravelled down to the atomic scale. The scientific concepts of the NG group are therefore based on applying and developing advanced electron microscopy techniques such as aberration-corrected (scanning) transmission electron microscopy ((S)TEM), energy-dispersive X-ray spectroscopy (EDX) and electron energy-loss spectroscopy (EELS). The atomistic structure, chemical composition, and bonding of material imperfections in bulk materials [48, 49] are investigated, as well as those in thin films [50-56] or individual nanostructures [57-67]. Own principal component analysis (PCA) tools are developed and applied to analyse the EDX and EELS data [40, 50, 68]. 3D morphology on nanostructures is characterized via electron tomography [1, 38-40], whereas their chemical composition in 3D is resolved by atom probe tomography (APT) [1, 6, 10]. The observed microstructural features (such as interfaces) or nanostructures are coupled to the functional properties (optical, electrical, thermal or electrochemical). This allows to establish property-nano/microstructure relationships, optimize material concepts, establish new synthesis pathways, or uncover beneficial (or detrimental) effects of dopants and impurities.

The stability of renewable energy materials is a pre-requisite for successful application and often overlooked. The NG group has a strong commitment to understand the origin of degradation phenomena and develops methodologies, which help to resolve them. For example, a light source was added to the existing scanning flow cell setup coupled to an inductively coupled plasma mass-spectrometer (iSFC-ICPMS) to explore the photostability of absorber materials (see p. 67). In brief, the material dissolution can be monitored on-line and correlated to *post-mortem* STEM/APT studies to observe the structural changes after photoelectrochemical cycling [4, 5, 72]. Further activities are based on an identical location (IL) TEM setup, which allows studying individual nanoparticles during electrochemical cycling and to uncover degradation phenomena. Thermal stability, an important issue for several renewable energy materials, is investigated by *in situ* electron microscopy heating experiments [32, 53, 69].

Following the scientific concepts, projects in the NG group cover a wide range of material systems. The thermoelectric materials are studied by L. Abdellaoui, R. Bueno Villoro, C. Jung, and S. Zhang. The materials for fuel cells are investigated by A. Garzón-Manjón, N. Rivas, M. Vega Paredes. R. Aymerich Armengol, R. Changizi, A. Frank, T. Gänsler, J. Lim, R. Sahu, and S. Zhang perform research on materials for (photo)-electrolysis, light emitting devices and energy storage. Studies related to metallic thin films with a focus on stability against hydrogen ingress, thermal load and corrosive attack are done by S. Zhang and S. Hieke (former member). These studies are supported by S. Reckort who is in charge of the TEM sample preparation laboratory. A recent picture from an online NG group meeting is presented in Fig. 1.

Fig. 1: *NG group member meeting in summer 2021.*

Supervision, teaching and mentoring

The sharing of knowledge via teaching and supervision is a central philosophy of the NG group. This is reflected in the active recruitment of bachelor and master students from German universities and abroad. Several of the members started either during their bachelor thesis or as student assistants and continued with their master and PhD theses. During the last 3 years, two bachelor, four master, and three PhD students graduated with great success. C. Scheu, S. Zhang and A. Garzón-Manjón are involved in teaching activities in universities and international schools. C. Scheu is a full professor at the RWTH Aachen University, and organizes courses in the Division of Materials Science and Engineering, as well as in the master curriculum at the Physics Faculty of Heinrich-Heine University in Düsseldorf. C. Scheu, S. Zhang and A. Garzón-Manjón serve as committee members in several university boards. C. Scheu also acts as co-supervisor of external PhD students from Ruhr-Universität Bochum (RUB), University of Duisburg-Essen (UDE), Ludwig-Maximilians-University (LMU) and RWTH Aachen University.

Supporting female scientists on their pathway to become a professor or a leader at an academic or industrial institution is a centrepiece of the mentoring program established by C. Scheu in 2018 at the MPIE. Currently, she is the mentor of 10 postdoctoral researchers at different career levels. Besides individual coaching, regular joint meetings with the mentees are held, with a strong support from S. Degner, M. Todorova and K. Hübel (see p. 99). A recent success of this action is the acceptance of all 4 mentees nominated by the MPIE for the highly competitive career building “Sign-Up 2022!” program of the Max Planck Society (MPG).

Funding and collaboration

Research funding is received from the Federal Ministry of Economic Affairs and Energy (BMWi) for the project PAULL, which is dedicated to investigate materials for fuel cells (see below). Funding for the project H2BS - barrier coatings against hydrogen ingress - is also provided by BMWi within the community industrial research (IGF) program (see p. 84). The project CARMON on battery materials was funded by the Leibniz Association (see p. 83). Financial support was also obtained from the German Research Foundation (DFG) either within priority programmes such as the SFB 1394 (start 01/2020) and SPP 1613 (until 04/2019) or within individual grants together with university partners (summary of the last three projects described later in section research examples). The ongoing SFB 1394 covers structural and chemical atomic complexity, defect phase diagrams to material properties in Mg-Al-Ca alloys (see p. 76). The NG group is contributing here by analysing the structure and composition of grain boundaries, phase boundaries and surfaces. Funding is also received from the MPG via the SURMAT international school (see p. 74).

The NG group is embedded in a national and international network of university partners and research institutions. This includes in Germany the RWTH Aachen University, RUB, LMU, UDE, University of Konstanz, Leibniz Institute for Plasma Science and Technology Greifswald (INP), the Leibniz Institute for New Materials Saarbrücken (INM), and the Helmholtz Center Hereon, Geesthacht. International partners come from the Czech Republic, China, France, Israel, Italy, South Korea, Spain and USA. In addition, the group maintains long-lasting partnerships with fuel cell industries.

Since the foundation of the NG group in 2014, strong collaborations were established with the MA, SN, GO and CM departments, which is documented in a large number of joint publications and supervised PhD students. There is also a close interaction with the recently established Max Planck Fellow Group on Electrochemistry & Nanoscale Materials (see p. 22).

Organized conferences and workshops

Due to the COVID-19 pandemic, new formats were established to interact with international colleagues. The 1st Virtual Thermoelectric Conference 2020 (VCT 2020) replaced the annual on-site meeting and attracted over 1000 participants. The European session was organized by L. Abdellaoui and members from the NG group and MA department with technical support by T. Sondermann (SN), B. Beckschäfer (MA), A. Kuhl (MA) and S.L. Wong (GWDG, IT service organization for the MPG and the University Göttingen). The success was continued in the 2nd Virtual Thermoelectric Conference 2021 (VCT 2021), with L. Abdellaoui and her team organizing the European session. Together with W. Kaplan (Technion, Israel) and J. Blendell (Purdue University, USA), C. Scheu started in autumn 2020 the monthly online event “International seminar series on the microstructure of materials” with around 60 participants. C. Scheu was the session chair of MS3, energy-related materials and catalysts at the Microscopy Conference (MC) 2019 in Berlin, and organized the IAMNano (International workshop on advanced and *in situ* microscopies of functional nanomaterials and devices) held in Düsseldorf in 2019 with G. Dehm, C. Liebscher, and B. Völker (all SN department). C. Scheu also co-organized the yearly joint retreat “Mechanics meets energy” between the SN department and the NG group in 2019 and 2020.

Dissemination and awards

The NG members gave invited talks at international conferences and colloquia in universities and research institutions either in Germany or abroad. A selection is given here. For example, L. Abdellaoui gave invited lectures at the 6th German-Korean Thermoelectric workshop in Daegu, South Korea (2019) and at the North American Thermoelectric workshop, Northwestern University, Chicago, USA (2019). S. Zhang had invited presentations at the BiGmax network event in Platja d’Aro, Spain (2019) and at the LMU in Munich, Germany (2020). R. Sahu presented invited speeches at the Microscopy workshop at IIT Madras (2020) and VIT Vellore, India (2021). C. Scheu presented on-site invited talks at the SCANDEM 2019 in Gothenburg, Sweden, at the ISMANAM-2019 in Chennai, India and at the Universita’ Roma Tre, in Roma, Italy (2019), and on-line invited talks at the international workshop on high entropy alloy and complex solid solution nanoparticles for electrocatalysis, organized by RUB (2020), the ICMCTF2021 virtual conference (2021), etc. Besides invited talks, all NG members present their projects at international conferences and workshops. Many received awards for their scientific works. For example, R. Changizi won the first prize of the best image award at the MC 2019, Berlin. R. Aymerich Armengol was selected to participate at the 70th Interdisciplinary Lindau Nobel Laureate meeting held online in June 2020 and June 2021. A. Garzón-Manjón was chosen by a selection committee to present a video on her research activities produced by Latest Thinking. C. Jung was awarded with a postdoctoral fellowship program grant from the Korean NRF (2021). R. Aymerich Armengol and R. Changizi won the MPIE science video competition. J. Lim, an Alexander von Humboldt fellow, was appointed as an assistant professor at Kangwon National University in South Korea. C. Scheu was awarded to be a panel member for the strategic evaluation of the research field "Matter" of the Helmholtz Association (till 2020) and of the review board "Production and processing of functional materials" of the DFG (2020).

Selected research examples

The following is a brief report on the scientific results of some of our research activities. Further projects are described in the sections on interdepartmental research activities - selected highlights, long term-oriented method developments and large-scaled and networking projects.

Thermoelectric materials

A promising way to recycle energy from waste heat is by the use of thermoelectric devices to generate electricity. The efficiency of such devices is governed by the dimensionless figure of merit *ZT*, which is proportional to the electrical conductivity, the Seebeck coefficient and temperature, and inversely proportional to the thermal conductivity. Microstructural features such as point defects, dislocations, planar defects, grain boundaries, etc. interact with charge carriers and phonons, and their effects on the electrical and thermal conductivities are crucial factors to design thermoelectric materials [11, 32-37].

In order to understand the impact of these defects, their number densities, structural and chemical characteristics are studied from the device scale down to the atomic scale. This scale-bridging methodology was applied by L. Abdellaoui, R. Bueno Villoro, C. Jung and S. Zhang for the first time to characterize stacking faults [34] and dislocations [32] in thermoelectric materials. SEM-based techniques including electron backscattered diffraction (EBSD) and electron channeling contrast imaging (ECCI) cover the mm to µm scale. Identified features are then extracted by focused ion beam (FIB) for (S)TEM microscopy and microanalysis down to the atomic scale, as well as APT for the analysis in 3D down to near-atomic resolution. Such correlative and extensive analysis is facilitated by close interactions between S. Zaefferer's group (MA department) with ECCI expertise and B. Gault's group (MA department), O. Cojocaru-Mirédin and Y. Yu (both RWTH Aachen University) with APT expertise.

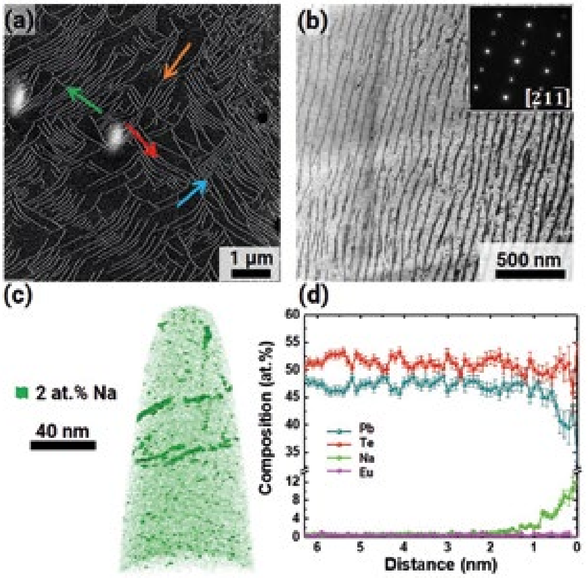


Fig. 2: *a) ECCI image showing four groups of dislocations along <111> directions. b) Bright field TEM image revealing a high density of dislocations (inset: selected area diffraction pattern along the* $[21\bar{1}]$ *zone axis). c) APT reconstruction demonstrating Na enrichment at the dislocations. Composition proximity histogram proving a Na Cottrell atmosphere at the dislocation core. Data from [32].*

As an example, it is commonly hypothesized that dislocations are responsible for reducing the thermal conductivity in many thermoelectrics. Nevertheless, in order to reach the ultra-low 0.4 W/m/K measured in $Eu_{0.03}Na_{0.025}Pb_{0.945}Te$, an unphysically high dislocation density of $4×10^{12}$ cm^{-2} is required in the phonon transport model as proposed by Y. Pei (Tongji University, China). In collaboration with him and his group, the scale-bridging methodology was applied to investigate the structural defects responsible for the ultralow thermal conductivity [32]. As shown in Fig. 2, the density of dislocations is only on the order of 10^{10} cm^{-2}, but they aligned to form 4 groups of parallel networks. ECCI at controlled diffraction conditions revealed that the 4 groups of dislocations are along $[\bar{1}\bar{1}1]$, $[\bar{1}1\bar{1}]$, $[\bar{1}11]$, and $[111]$ and directions, as marked in blue, green, red and orange, respectively. TEM analysis confirmed the edge character of the dislocations. As a result of the parallel alignments, the strain fields of dislocations superimpose and scatter phonons across the range of an entire grain, 10 µm on average, rather than the dislocation spacing, 0.1 µm, leading to a remarkable reduction in thermal conductivity [32]. Moreover, the chemical composition of the dislocations was determined using APT. Na Cottrell atmospheres were found around dislocation cores (Fig. 2c). Cottrell atmospheres and their introduced strain field increase the phonon scattering cross-sections of dislocations by another factor of 2~5 [11, 32]. Overall, both the large-scale alignment and atomic-scale segregation of dislocations have major implication on thermal conductivity, and such scale-bridging characterization is indispensable to understand transport properties of thermoelectrics.

Dislocations not only reduce the thermal conductivity of materials, but they also increase their mechanical strengths. In a collaborative work with G. J. Snyder's group (Northwestern University, USA) [33], a correlation between the dislocation densities and the brittleness in PbTe-based thermoelectric materials was uncovered. Some auspicious roads towards suppressing brittleness while maintaining the high thermoelectric performance enabled by defect engineering are important for future developments of p- and n-type PbTe[33] .

Furthermore, in practical applications, thermoelectric generators operate over large temperature gradients to profit from a high Carnot efficiency. It is therefore important to investigate how the microstructures designed for room temperature evolve at high temperature. The NG group performs *in situ* heating experiments on thin TEM lamellae of thermoelectrics to track their microstructural evolution. The lamellae were prepared by FIB and welded onto micro-electromechanical system (MEMS) heating chips (Fig. 3a, b). For example, a high thermal stability of the aforementioned dislocation networks of Na-doped PbTe in STEM was observed (Fig. 3c) [32].

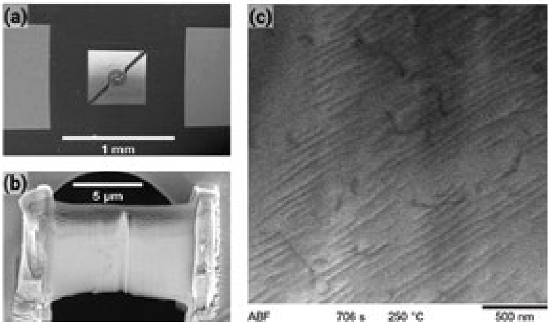


Fig. 3: *a) In situ heating chip with b) FIB-prepared sample. c) An excerpt from in situ heating video showing dislocation networks in an* $Eu_{0.03}Na_{0.025}Pb_{0.945}Te$ *alloy which are immobile during 250 °C isothermal annealing. Data from [32].*

Moreover, the formation and dissolution of precipitates in PbTe-based thermoelectrics is observed at different temperatures (manuscripts under preparation). A practical limit for the *in situ* heating experiments is the sublimation of PbTe at elevated temperatures under the TEM vacuum. As an outlook, *in situ* heating will be conducted in an environmental TEM to extend the experiments to higher temperatures and longer durations.

The electrical conductivity plays also an important role in enhancing *ZT* [35, 37]. Joint activities with H. Bishara (SN department) include local electrical conductivity measurements to determine the influence of defects such as grain boundaries. An example for NbCo(Pt)Sn Half-Heusler compounds [35] is described on p. 188.

Materials for fuel cells

In order to lessen climate change induced by the transportation sector, electrified cars, trucks and ships need to be developed. Hydrogen is used as fuel to generate electricity via polymer electrolyte membrane fuel cells (PEMFCs). The NG group dedicates efforts in improving the life-time and stability of PEMFCs by studying the core components before and after operation. Structural analysis from entire components down to the atomic scale allows to understand degradation, a prerequisite for improved materials design. In the recently finished PhD thesis of the external NG member M. Obermaier, the degradation of automotive PEMFCs was studied [25-27]. A mechanistic understanding was achieved, which can explain the local fuel starvation degradation of an automotive PEMFC full size stack [27].

One of the bottlenecks for the widespread commercialization of PEMFCs is the Pt catalyst, in particular the high cost and low tolerance to impurities. Right now, Pt is used for both the oxygen reduction reaction (ORR) on the cathode and the hydrogen oxidation reaction (HOR) on the anode. However, the Pt catalysts for HOR can be poisoned by impurities in the hydrogen feedstock, e.g. carbon monoxide (CO). A successful approach for mitigating those hurdles is based on Pt-Ru catalysts. Their long-term stability still needs to be improved, and the challenge is tackled in close industrial cooperation as part of the BMWi project "PAULL". Novel Ru@Pt core-shell nanoparticles (NPs) are explored as catalysts in place of traditional Pt-Ru solid solutions.

PEMFCs with Ru@Pt core-shell NPs on the anode and pure Pt at the cathode were subjected to accelerated degradation tests. The functional layers of the membrane electrode assembly thinned down during the degradation test but were still intact as demonstrated by SEM investigations. TEM revealed that Ostwald ripening, aggregation and dissolution processes of the catalyst occurred, so that NPs grew in size at the electrodes and formed aggregated bands in the membrane (Fig. 4a). Such NP bands were also observed after operation of PEMFCs using Pt-Ru solid solution NPs as catalysts, with 50 at% composition of Ru and Pt. As seen in Fig. 4a, the NP band is located near the cathode-membrane interface. From STEM (Fig. 4b) and EDS analysis (Fig. 4c), the NP particles were identified to possess a fcc crystal structure with little Ru present. In general, a lower amount of Ru was found in the band when using Ru@Pt catalysts (<10 at.%) compared to the ones using solid solution NPs. The results confirm the protection effect of the Pt shell on the Ru core and hence a superior degradation resistance of Ru@Pt NPs compared to Pt-Ru solid solution NPs.

One of the characterization techniques that is currently being implemented for the fuel cell research is correlative TEM-APT experiments. First results reveal the presence of Cl impurities within the NP, which are due to the precursor and might be detrimental for their catalytic activity.

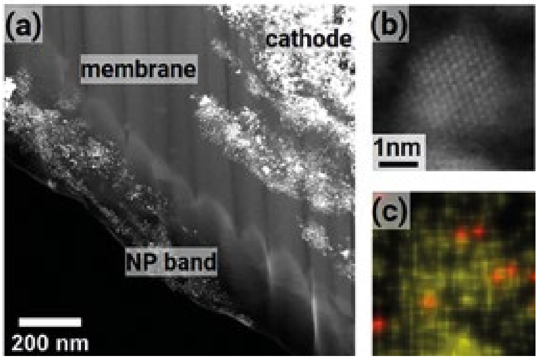


Fig. 4: *a) High-angle annular dark-field (HAADF) STEM micrograph showing a metallic band next to cathode-membrane interface after accelerated stress test. b) STEM and c) EDX of a selected NP within the band. Red colour represents Ru, yellow colour Pt.*

Besides catalysts for PEMFCs which operate in acidic conditions, the NG group is also interested in catalysts for alkaline anion exchange membrane fuel cells. For these type of fuel cells, it is crucial to find novel efficient ORR catalysts to promote the kinetically-limiting ORR. Together with the groups of A. Ludwig and W. Schuhmann (both at RUB) it was discovered that 1-3 nm sized NPs consisting of earth-abundant transition metal elements, namely the Cantor alloy (CrMnFeCoNi), show a high intrinsic ORR activity [28-31] in alkaline media. Direct current sputtering from either five elemental targets or one alloy target is used to synthesize these complex solid solution (CSS) NPs within ionic liquids. Ionic liquids (salts with a melting point < 100 °C) have low vapor pressures and are therefore suitable substrates for the sputtering process. In addition, the ionic liquid itself can act as an electronic and steric stabilizer preventing nanoparticle growth and aggregation, hence, leading to the formation of NPs with tunable structures and compositions [28-31]. This work was partially funded by the DFG within the grant SCHE634/21 1.

Electron microscopy performed by A. Garzón-Manjón was the key in understanding the excellent intrinsic ORR activity of the CSS NPs and allowed to establish structure-activity relationships needed for targeted catalyst design. With the help of aberration-corrected (S)TEM and EDS mapping the crystallinity and chemical composition of the NPs were identified. Novel pathways to produce CSS NPs with different sizes (from 1.3 ± 0.1 nm up to 2.6 ± 0.3 nm), crystallinity (amorphous, face-centred cubic (fcc) or body-centred cubic (bcc)) and composition (e.g., from 19 at.% to 2 at% Mn) using a library of eight ionic liquids were developed [30]. The different CSS NPs showed high activity towards the ORR in alkaline media. The highest activity was found for NPs with a bcc structure, lower Mn and Fe contents, and higher Cr and Co contents, as synthesized using the ionic liquid [Emimi][(Tf)$_2$N] as substrate [30].

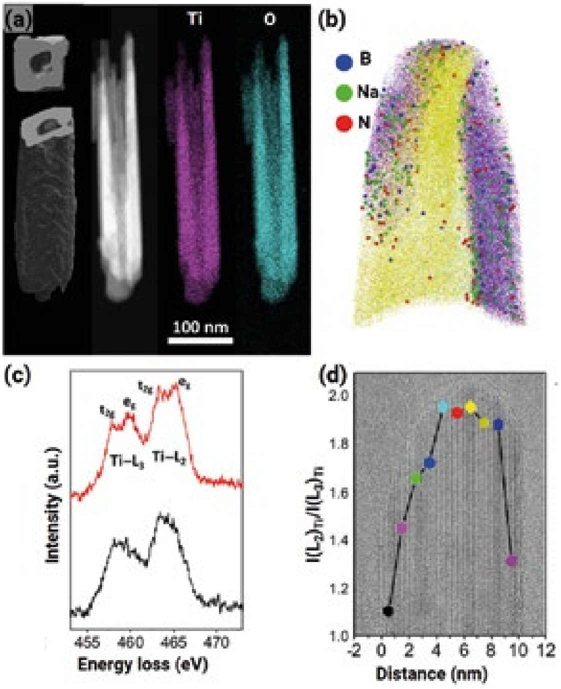


Fig. 5: *a) Electron tomographic reconstruction and EELS elemental maps of a hollow, black* TiO_2 *nanowire. b) APT reconstruction showing the enrichment of B, Na and N at the* TiO_2 *surface. c) ELNES of* $Ti\text{-}L_{2,3}$ *edge revealing the presences of* Ti^{4+} *and* Ti^{3+} *ions. d)HRTEM image superimposed with the* L_2/L_3 *white line ratio (a low value corresponds to a high number of* Ti^{3+} *ions). Data from [1].*

In order to investigate structural evolution of the CSS NPs at elevated temperatures, a CrMnFeCoNi alloy target was directly sputtered on a SiN_x-based MEMS heating device (Fig. 3a). The as-sputtered film was separated into two phases: bcc CoNi-rich nanoscale islands and a continuous bcc CrMnFe-rich layer, indicating a Stranski–Krastanov growth process [70]. *In situ* heating TEM experiments of this CSS nanostructure revealed the phase stability up to 700 °C, where Ostwald ripening took place leading to abnormal growth of NPs and accompanied changes in chemical composition [70]. Studies on continuous fcc (Mn)CrFeCoNi films [55, 56] are given in the section large scale networks/projects, p. 78.

Nanostructured oxides and sulphides for (photo) electrochemical, optical and storage application

Nanostructured oxides are successfully employed as (photo)electrocatalyst, catalyst support and battery materials. Their properties are governed by their large surface-to-volume ratio, which allows to achieve a high activity. One of the most widely studied nanostructured materials is TiO_2, yet, new aspects are still discovered. To extend the light absorption well into the visible range, defects such as oxygen vacancies are introduced to make black TiO_2, named after its dark colour. In a recent work of J. Lim and R. Aymerich Armengol, black TiO_2 hollow nanowires were synthesized via a hydrothermal approach and subsequent reduction at high temperatures using $NaBH_4$ [1]. Their morphology is shown in the electron tomogram and the distribution of the major elements in the EELS maps in Fig. 5a. Impurities were detected by APT as discussed below (Fig. 5b). The analysis of the energy loss near-edge structure (ELNES) of the Ti $L_{2,3}$ edges (Fig. 5c) showed that the Ti ions at the surface are in a reduced Ti^{3+} oxidation state, in line with a higher number of O vacancies compared to the bulk interior where Ti^{4+} states dominate (Fig. 5d). However, black TiO_2 is known to be unstable in air, as Ti^{3+} gets oxidized in the atmosphere. To investigate how Ti^{3+} could be stabilized, the hollow nanowires were analysed by APT in collaboration with the group of B. Gault. As shown in Fig. 5b, N, B and Na impurities were found on the surface of the nanowires. They alter the surface leading to a distorted rutile structure and stabilize the large number of oxygen vacancies and Ti^{3+} ions, which govern the electrochemical performance as tested together with O. Kasian (GO department). The N, B, and Na impurities stem from the reducing atmosphere, the reducing agent and the glass substrate, respectively [1]. In addition to increased visible absorption, the black TiO_2 nanowires also have higher electrical conductivity important for their electrochemical applications [1]. The black TiO_2 hollow nanowires were applied as a support for Pt catalysts and tested with M. Ledendecker (GO department) in acidic conditions [2]. The formation of a TiO_2 encapsulation layer around Pt nanoparticles was observed, which changes the electrochemical response. Despite widely regarded as a stable oxide, the formation of an encapsulation layer shows that the TiO_2 was not stable under the chosen condition and it also changed the average oxidation state. Ongoing studies by S. Zhang using the iSFC-ICPMS setup revealed also the dissolution of TiO_2 under photoelectrochemical conditions.

The iSFC-ICPMS setup developed in the NG group was applied to investigate the photocorrosion mechanisms in highly photoactive materials such as $BiVO_4$ [4, 5 and p. 67]. Within the DFG SPP 1613 on regeneratively produced fuels by light driven water splitting, the NG group contributed in establishing microstructure-activity-stability relationships for various (photo)anode materials, including IrO_2 [20, 23], Fe_2O_3 [10, 11], TiO_2 [21], $BiVO_4$, [5, 14, 17], $MgFe_2O_4$ [18] and on the cathode side, Cu_2O [15], NiO_x [71] and $(Al,Cr,Fe)O_4$ [12]. Another project related to water splitting is the collaborative work with B. Lotsch (Max Planck Institute for Solid State Research), where it was found that strain in a Pd-rich capping layer of $PdCoO_2$ enhances the HER in acidic media [7]. Studies with the group of A. Llobet (Institute of Chemical Research of Catalonia, Spain), who synthesized novel catalysts for the water oxidation reaction, which are based on Ru coordination oligomers adsorbed on multiwalled carbon nanotubes, were conducted to prove the presence of Ru [7].

Nb_3O_7OH is another known photocatalytically active material, where in-depth TEM investigations are performed by T. Gänsler to understand the growth of nanostructures and their behaviour. Defects play a central role and are also analysed to explain the complementary resistive switching behaviour [57, 58] found in Nb_3O_7OH and TiO_2 within the collaborative DFG project SCHE 634/20-1 with L. Schmidt-Mende (Konstanz University). Fig. 6 shows an aberration corrected HRTEM image revealing the presence of planar defects but also resolving the arrangement of the surface atoms. This is further elucidated with the help of density functional theory calculations of J. Minar (University of West Bohemia, Plzen, Czech Republic). Nb_3O_7OH can be transformed to Nb_2O_5 by the release of water, the latter is an interesting battery material. Earlier *in situ* heating TEM experiments resulted in the transformation from Nb_3O_7OH to NbO, as lattice oxygen was lost in the TEM vacuum [72]. Recent environmental TEM experiments performed by S. Betzler (Lawrence Berkeley National Laboratory, USA) in collaboration with B. Sinclair (Stanford University, USA) allowed to study the reaction from NbO to T-Nb_2O_5 at elevated temperatures when oxygen is supplied [3]. A partial oxygen pressure of <1mbar is already sufficient. The reaction is in accordance to the bulk phase diagram and starts at the planar faults within the wires [3].

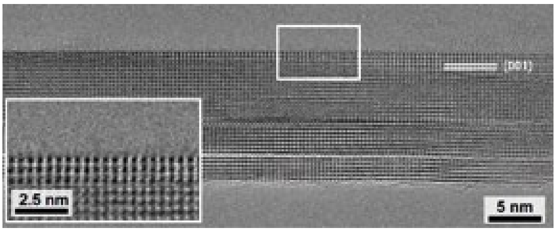


Fig. 6: *HRTEM micrograph of a Nb_3O_7OH nanowire showing the presence of planar defects and the surface structure (magnified shown as inset).*

Crystal defects have also considerable effects on the optical properties. For example, individual $PrNbO_4$ and $Pr^{3+}:Ca_2Nb_2O_7$ particles were studied with cathodoluminescence (CL) measurements in an SEM by R. Changizi, who correlated the findings with the structural and chemical information obtained via (S)TEM and EDS [47]. The lanthanide ions' surroundings, concentration and presence of defects such as twin boundaries determine the width and intensity of the emission lines. The effect of the twin density on the optical properties of $PrNbO_4$ is being studied in close collaboration with S. Zaefferer (MA department). High density twinned particles show peaks with higher intensity compared to the twin-free particles. Presence of the twin boundaries leads to the hybridization of the 4f electronic states, which enables the otherwise parity-forbidden inner 4f-shell transitions.

2D nanosheets are another interesting class of materials for (photo)electrocatalysis. S. Zhang explored the photocatalytic $HCa_2Nb_3O_{10}$ nanosheets as the support for co-catalysts, for example, NiO_x for the hydrogen evolution reaction [69]. Uniform thickness of the nanosheets facilitated the TEM observation on their structural evolution

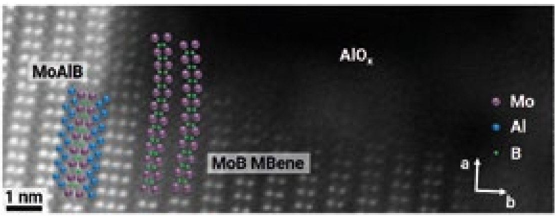


Fig. 7: *Aberration-corrected STEM image revealing the local formation of MoB MBene layers in MoAlB adjacent to amorphous AlO_x region. Data taken from [50].*

(see p. 65). R. Aymerich Armengol and J. Lim synthesized $Fe_xMn_{1-x}O_2$ 2D nanosheets with different doping amounts using a facile novel wet-chemical procedure. These nanosheets catalyse very efficiently the growth of carbon nanotubes at low temperatures [45]. Another 2D material, MBene, was fabricated by direct synthesis of MoAlB films via DC magnetron sputtering using a moderate growth temperature of 700 °C in collaboration with J. M. Schneider (RWTH Aachen University) [50]. The work by R. Sahu reveals that 2D MBene formation occurs by Al deintercalation from MoAlB in the vicinity of AlO_x without any external stimuli such as chemical etching or heat treatment. Hence, it is a straightforward method of MoB MBene-AlO_x heterostructures synthesis [50]. The resulting structure is shown in Fig. 7. The material might possess potential for energy storage and electrocatalytic application.

More examples where the NG group is involved in the design and understanding of electrocatalysts can be found in p. 184. The studies related to battery materials are presented in the project CARMON (see p. 83).

References

1. Lim, J.; Kim, S.H.; Armengol, R.A.; Kasian, O.; Choi, P.P.; Stephenson, L.T.; Gault, B.; Scheu, C.: Angew. Chem. - Int. Ed. 59 (2020) 5651.
2. Armengol, R.A.; Lim, J.; Ledendecker, M.; Hengge, K.; Scheu, C.: Nanoscale Adv. 3 (2021) 5075.
3. Betzler, S.B.; Koh, A.L.; Lotsch, B.V.; Sinclair, R.; Scheu, C.: ACS Appl. Nano Mater. 3 (2020) 9285.
4. Zhang, S.Y.; Ahmet, I.; Kim, S.H.; Kasian, O.; Mingers, A.M.; Schnell, P.; Kolbach, M.; Lim, J.; Fischer, A.; Mayrhofer, K.J.J.; Cherevko, S.; Gault, B.; van de Krol, R.; Scheu, C.: ACS Appl. Energy Mater. 3 (2020) 9523.
5. Zhang, S.Y.; Rohloff, M.; Kasian, O.; Mingers, A.M.; Mayrhofer, K.J.J.; Fischer, A.; Scheu, C.; Cherevko, S.: J. Phys. Chem. C 123 (2019) 23410.
6. Kim, S.H.; Lim, J.; Sahu, R.; Kasian, O.; Stephenson, L.T.; Scheu, C.; Gault, B.: Adv. Mater. 32 (2020) 1907235.
7. Podjaski, F.; Weber, D.; Zhang, S.Y.; Diehl, L.; Eger, R.; Duppel, V.; Alarcon-Llado, E.; Richter, G.; Haase, F.; Morral, A.F.I.; Scheu, C.; Lotsch, B.V.: Nat. Catal. 3 (2020) 55.
8. Kampmann, J.; Betzler, S.; Hajiyani, H.; Haringer, S.; Beetz, M.; Harzer, T.; Kraus, J.; Lotsch, B.V.; Scheu, C.; Pentcheva, R.; Fattakhova-Rohlfing, D.; Bein, T.: Nanoscale 12 (2020) 7766.
9. Böhm, D.; Beetz, M.; Kutz, C.; Zhang, S.Y.; Scheu, C.; Bein, T.; Fattakhova-Rohlfing, D.: Chem. Mater. 32 (2020) 10394.
10. Hufnagel, A.G.; Hajiyani, H.; Zhang, S.Y.; Li, T.; Kasian, O.; Gault, B.; Breitbach, B.; Bein, T.; Fattakhova-Rohlfing, D.; Scheu, C.; Pentcheva, R.: Adv. Funct. Mater. 28 (2018) 1804472.
11. Yu, Y.; Zhou, C.J.; Zhang, S.Y.; Zhu, M.; Wuttig, M.; Scheu, C.; Raabe, D.; Snyder, G.J.; Gault, B.; Cojocaru-Miredin, O.: Mater. Today 32 (2020) 260.
12. Stein, H.S.; Zhang, S.Y.; Li, Y.J.; Scheu, C.; Ludwig, A.: Z. Phys. Chem. 234 (2020) 605.
13. Lukic, S.; Busser, G.W.; Zhang, S.Y.; Menze, J.; Muhler, M.; Scheu, C.; Winterer, M.: Z. Phys. Chem. 234 (2020) 1133.
14. Rohloff, M.; Anke, B.; Wiedemann, D.; Ulpe, A.C.; Kasian, O.; Zhang, S.Y.; Scheu, C.; Bredow, T.; Lerch, M.; Fischer, A.: Z. Phys. Chem. 234 (2020) 655.
15. Yang, F.; Schrock, C.; Kugelstadt, J.; Zhang, S.Y.; Scheu, C.; Trautmann, C.; Maijenburg, W.; Toimil-Molares, M.E.: Z. Phys. Chem. 234 (2020) 1205.
16. Kumari, S.; Helt, L.; Junqueira, J.R.C.; Kostka, A.; Zhang, S.Y.; Sarker, S.; Mehta, A.; Scheu, C.; Schuhmann, W.; Ludwig, A.: Int. J. Hydrogen Energy 45 (2020) 12037.
17. Rohloff, M.; Anke, B.; Kasian, O.; Zhang, S.Y.; Lerch, M.; Scheu, C.; Fischer, A.: ACS Appl. Mater. Interfaces 11 (2019) 16430.
18. Timm, J.; Bloesser, A.; Zhang, S.Y.; Scheu, C.; Marschall, R.: Microporous Mesoporous Mater. 293 (2020) 109783.
19. Ledendecker, M.; Geiger, S.; Hengge, K.; Lim, J.; Cherevko, S.; Mingers, A.M.; Gohl, D.; Fortunato, G.V.; Jalalpoor, D.; Schuth, F.; Scheu, C.; Mayrhofer, K.J.J.: Nano Res. 12 (2019) 2275.
20. da Silva, G.C.; Venturini, S.; Zhang, S.Y.; Loeffler, M.; Scheu, C.; Mayrhofer, K.J.J.; Ticianelli, E.A.; Cherevko, S.: Chemelectrochem. 7 (2020) 2330.
21. Sahin, E.O.; Dai, Y.T.; Chan, C.K.; Tuysuz, H.; Schmidt, W.; Lim, J.; Zhang, S.Y.; Scheu, C.; Weidenthaler, C.: Chem. - Eur. J. 27 (2021) 11600.
22. Sahin, E.O.; Tuysuz, H.; Chan, C.K.; Moon, G.H.; Dai, Y.T.; Schmidt, W.; Lim, J.; Scheu, C.; Weidenthaler, C.: Nanoscale 13 (2021) 150.
23. Kasian, O.; Geiger, S.; Li, T.; Grote, J.P.; Schweinar, K.; Zhang, S.Y.; Scheu, C.; Raabe, D.; Cherevko, S.; Gault, B.; Mayrhofer, K.J.J.: Energy Environ. Sci. 12 (2019) 3548.
24. Hoque, M.A.; Gil-Sepulcre, M.; de Aguirre, A.; Elemans, J.; Moonshiram, D.; Matheu, R.; Shi, Y.Y.; Benet-Buchholz, J.; Sala, X.; Malfois, M.; Solano, E.; Lim, J.; Garzón-Manjón, A.; Scheu, C.; Lanza, M.R.; Maseras, F.; Gimbert-Surinach, C.; Llobet, A.: Nat. Chem. 12 (2020) 1060.
25. Obermaier, M.; Jozwiak, K.; Rauber, M.; Bauer, A.; Scheu, C.: J. Power Sources 488 (2021) 229405.
26. Obermaier, M.; Bauer, A.; Dalkilic, M.; Rauber, M.; Scheu, C.: Fuel Cells 21 (2021) 221.
27. Obermaier, M.; Rauber, M.; Bauer, A.; Lochner, T.; Du, F.; Scheu, C.: Fuel Cells 20 (2020) 394.
28. Löffler, T.; Savan, A.; Garzón-Manjón, A.; Meischein, M.; Scheu, C.; Ludwig, A.; Schuhmann, W.: ACS Energy Lett. 4 (2019) 1206.
29. Löffler, T.; Meyer, H.; Savan, A.; Wilde, P.; Garzón-Manjón, A.; Chen, Y.T.; Ventosa, E.; Scheu, C.; Ludwig, A.; Schuhmann, W.: Adv. Energy Mater. 8 (2018) 1802269.
30. Garzón-Manjón, A.; Löffler, T.; Meischein, M.; Meyer, H.; Lim, J.; Strotkotter, V.; Schuhmann, W.; Ludwig, A.; Scheu, C.: Nanoscale 12 (2020) 23570.
31. Garzón-Manjón, A.; Meyer, H.; Grochla, D.; Löffler, T.; Schuhmann, W.; Ludwig, A.; Scheu, C.: Nanomater. 8 (2018) 903.
32. Abdellaoui, L.; Chen, Z.W.; Yu, Y.; Luo, T.; Hanus, R.; Schwarz, T.; Villoro, R.B.; Cojocaru-Miredin, O.; Snyder, G.J.; Raabe, D.;

Pei, Y.Z.; Scheu, C.; Zhang, S.Y.: Adv. Funct. Mater. 31 (2021) 2101214.

33. Male, J.P.; Abdellaoui, L.; Yu, Y.; Zhang, S.Y.; Pieczulewski, N.; Cojocaru-Miredin, O.; Scheu, C.; Snyder, G.J.: Adv. Funct. Mater. (2021) 2108006.
34. Abdellaoui, L.; Zhang, S.Y.; Zaefferer, S.; Bueno-Villoro, R.; Baranovskiy, A.; Cojocaru-Miredin, O.; Yu, Y.; Amouyal, Y.; Raabe, D.; Snyder, G.J.; Scheu, C.: Acta Mater. 178 (2019) 135.
35. Luo, T.; Serrano-Sanchez, F.; Bishara, H.; Zhang, S.Y.; Villoro, R.B.; Kuo, J.J.; Felser, C.; Scheu, C.; Snyder, G.J.; Best, J.P.; Dehm, G.; Yu, Y.; Raabe, D.; Fu, C.G.; Gault, B.: Acta Mater. 217 (2021) 117147.
36. Gomell, L.; Katnagallu, S.; Diack-Rasselio, A.; Maier, S.; Perriere, L.; Scheu, C.; Alleno, E.; Gault, B.: Scr. Mater. 186 (2020) 370.
37. Gomell, L.; Roscher, M.; Bishara, H.; Jägle, E.A.; Scheu, C.; Gault, B.: Scr. Mater. 193 (2021) 153.
38. Lim, J.; Kasiri, G.; Sahu, R.; Schweinar, K.; Hengge, K.; Raabe, D.; La Mantia, F.; Scheu, C.: Chem. - Eur. J. 26 (2020) 4917.
39. Um, J.H.; Lim, J.; Hengge, K.; Scheu, C.; Yoon, W.S.; Lee, J.K.; Sung, Y.E.: Composites Part B 166 (2019) 613.
40. Frank, A.; Gänsler, T.; Hieke, S.; Fleischmann, S.; Husmann, S.; Presser, V.; Scheu, C.: Nanoscale Adv. 3 (2021) 1067.
41. Husmann, S.; Budak, O.; Quade, A.; Frank, A.; Kruth, A.; Scheu, C.; Tolosa, A.; Presser, V.: J. Power Sources 450 (2020) 227674.
42. Fleischmann, S.; Dorr, T.S.; Frank, A.; Hieke, S.W.; Doblas-Jimenez, D.; Scheu, C.; de Oliveira, P.W.; Kraus, T.; Presser, V.: Batteries Supercaps 2 (2019) 668.
43. Bodnar, W.; Schiorlin, M.; Frank, A.; Schulz, T.; Wohrl, N.; Miron, C.; Scheu, C.; Kolb, J.F.; Kruth, A.: Appl. Surf. Sci. 514 (2020).
44. Budak, O.; Srimuk, P.; Tolosa, A.; Fleischmann, S.; Lee, J.; Hieke, S.W.; Frank, A.; Scheu, C.; Presser, V.: Batteries Supercaps 2 (2019) 74.
45. Lim, J.; Jin, X.Y.; Hwang, S.J.; Scheu, C.: Adv. Funct. Mater. 30 (2020) 2003849.
46. Drews, M.; Buttner, J.; Bauer, M.; Ahmed, J.; Sahu, R.; Scheu, C.; Vierrath, S.; Fischer, A.; Biro, D.: Chemelectrochem.; 10.1002/celc.202101174 (2021).
47. Changizi, R.; Zhang, S.Y.; Ziegler, C.; Schwarz, T.; Lotsch, B.V.; Scheu, C.: ACS Appl. Electron. Mater. 3 (2021) 461.
48. Chang, Y.H.; Zhang, S.Y.; Liebscher, C.H.; Dye, D.; Ponge, D.; Scheu, C.; Dehm, G.; Raabe, D.; Gault, B.; Lu, W.J.: Scr. Mater. 178 (2020) 39.
49. Peter, N.J.; Gerlitzky, C.; Altin, A.; Wohletz, S.; Krieger, W.; Tran, T.H.; Liebscher, C.H.; Scheu, C.; Dehm, G.; Groche, P.; Erbe, A.: Materialia 7 (2019) 100396.
50. Sahu, R.; Bogdanovski, D.; Achenbach, J.O.; Zhang, S.Y.; Hans, M.; Primetzhofer, D.; Schneider, J.M.; Scheu, C.: Nanoscale 13 (2021) 18077.
51. Achenbach, J.O.; Sahu, R.; Völker, B.; Hans, M.; Primetzhofer, D.; Miljanovic, D.J.; Scheu, C.; Schneider, J.M.: Coatings 9 (2019) 510.
52. Stelzer, B.; Chen, X.; Bliem, P.; Hans, M.; Völker, B.; Sahu, R.; Scheu, C.; Primetzhofer, D.; Schneider, J.M.: Sci. Rep. 9 (2019) 8266.
53. Hieke, S.W.; Willinger, M.G.; Wang, Z.J.; Richter, G.; Chatain, D.; Dehm, G.; Scheu, C.: Acta Mater. 165 (2019) 153.
54. Frank, A.; Dias, M.; Hieke, S.; Kruth, A.; Scheu, C.: Thin Solid Films 722 (2021) 138574.
55. Kini, M.K.; Lee, S.; Savan, A.; Breitbach, B.; Addab, Y.; Lu, W.J.; Ghidelli, M.; Ludwig, A.; Bozzolo, N.; Scheu, C.; Chatain, D.; Dehm, G.: Surf. Coat. Technol. 410 (2021) 126945.
56. Addab, Y.; Kini, M.K.; Courtois, B.; Savan, A.; Ludwig, A.; Bozzolo, N.; Scheu, C.; Dehm, G.; Chatain, D.: Acta Mater. 200 (2020) 908.
57. Ebenhoch, C.; Gänsler, T.; Schupp, S.; Hagner, M.; Frank, A.; Scheu, C.; Schmidt-Mende, L.: APL Mater. 9 (2021) 071105.
58. Ebenhoch, C.; Kalb, J.; Lim, J.; Seewald, T.; Scheu, C.; Schmidt-Mende, L.: ACS Appl. Mater. Interfaces 12 (2020) 23363.
59. Kalb, J.; Folger, A.; Zimmermann, E.; Gerigk, M.; Trepka, B.; Scheu, C.; Polarz, S.; Schmidt-Mende, L.: Surf. Interfaces 15 (2019) 141.
60. Kalb, J.; Folger, A.; Scheu, C.; Schmidt-Mende, L.: J. Cryst. Growth 511 (2019) 8.
61. Burratti, L.; De Matteis, F.; Francini, R.; Lim, J.; Scheu, C.; Prosposito, P.: Appl. Sci. 11 (2021) 3470.
62. Burratti, L.; Ciotta, E.; Bolli, E.; Kaciulis, S.; Casalboni, M.; De Matteis, F.; Garzón-Manjón, A.; Scheu, C.; Pizzoferrato, R.; Prosposito, P.: Colloids Surf. A 579 (2019) 123634.
63. Schiesaro, I.; Burratti, L.; Meneghini, C.; Fratoddi, I.; Prosposito, P.; Lim, J.; Scheu, C.; Venditti, I.; Iucci, G.; Battocchio, C.: J. Phys. Chem. C 124 (2020) 25975.
64. Meischein, M.; Garzón-Manjón, A.; Frohn, T.; Meyer, H.; Salomon, S.; Scheu, C.; Ludwig, A.: ACS Comb. Sci. 21 (2019) 743.
65. Schmitz, A.; Meyer, H.; Meischein, M.; Garzón-Manjón, A.; Schmolke, L.; Giesen, B.; Schlusener, C.; Simon, P.; Grin, Y.; Fischer, R.A.; Scheu, C.; Ludwig, A.; Janiak, C.: RSC Adv. 10 (2020) 12891.
66. Giesen, B.; Nickel, A.C.; Manjón, A.G.; Toscano, A.V.; Scheu, C.; Kahlert, U.D.; Janiak, C.: J. Inorg. Biochem. 203 (2020) 110952.
67. Abuayyash, A.; Ziegler, N.; Meyer, H.; Meischein, M.; Sengstock, C.; Moellenhoff, J.; Rurainsky, C.; Heggen, M.; Garzón-Manjón, A.; Scheu, C.; Tschulik, K.; Ludwig, A.; Köller, M.: Nanomedicine 24 (2020) 102126.
68. Zhang, S.; Scheu, C.: Microscopy 67 (2018) i133.
69. Zhang, S.Y.; Diehl, L.; Wrede, S.; Lotsch, B.V.; Scheu, C.: Catalysts 10 (2020) 13.
70. Garzón-Manjón, A.; Zhang, S.; Völker, B.; Meischein, M.; Ludwig, A.; Scheu, C.: MRS Bulletin (2021) accepted.
71. Knoppel, J.; Zhang, S.Y.; Specle, F.D.; Mayrhofer, K.J.J.; Scheu, C.; Cherevko, S.: Electrochem. Commun. 96 (2018) 53.
72. Betzler, S.B.; Harzer, T.; Ciston, J.; Dahmen, U.; Dehm, G.; Scheu, C.: Cryst. Growth Des. 16 (2016) 4309.

Max Planck fellow group: Self-reporting materials

Group head: J. M. Schneider

Materials Chemistry, RWTH Aachen University

1 October 2015 to 30 September 2020

Future technology challenges will no longer be simply addressed by today's materials and processing solutions, which are often based on trial and error. Instead, guidance will be attained from correlative experimental and theoretical research bridging all length scales. It is e.g. evident that future engineering components will be made of materials that are self-reporting. Self-reporting materials "communicate" damage of the material via changes of properties that can be measured in service as a consequence of chemical changes on the atomic length scale and/or phase transformations. Harnessing this would provide invaluable functionality for damage assessment and control and would constitute a major leap forward as the "health" of any material component exposed to mechanical loads could be monitored in service. This fascinating basic materials science with tremendous technological implications was the focus of Schneider's Max Planck Fellow Group.

One material property that can be measured in service non-destructively is the electrical resistivity. Within the Max Planck Fellow Group, the resistivity changes of magnetron sputtered, amorphous Cr_2AlC thin films were measured during heating in vacuum for the first time. For this Pt electrodes were deposited onto the as-deposited Cr_2AlC thin films. Annealing amorphous Cr_2AlC first leads to the formation of a hexagonal disordered solid solution which subsequently transforms into the (ordered) hexagonal MAX phase. In our proof of concept study [1], it was shown that these phase changes can be revealed by *in situ* measurements of thermally induced resistivity changes.

After it was successfully proven that the *in situ* tracking of phase changes is possible, the self-reporting capabilities were extended to *in situ* tracking the oxidation of TiN, serving as a model system for hard coatings in industrial applications [2]. Fig. 1 shows *in situ* measured sheet resistances of TiN thin films at different temperatures plotted over the oxidation time and the remaining TiN thickness dependent on the oxidation time as calculated from the measured sheet resistances via the Van der Pauw equation. Diamonds in the bottom centre graph indicate remaining TiN film thickness as measured by *ex situ* scanning transmission electron microscopy (STEM). It is evident, that for increasing oxidation times and temperatures, the sheet resistance is increasing accordingly to the degradation of the residual TiN film. For the films annealed 4 h at 600 and 650 °C the residual thicknesses were predicted to be 0.590 and 0.078 µm by *in situ* sheet resistance measurements, respectively. The via *ex situ* STEM measured thicknesses were deviating by 8.3 % and 14.2 % from these values, respectively, and are hence in good agreement. For the sample annealed 1 h 25 min at 700 °C both the *in situ* and *ex situ* measurement consistently indicate the complete loss of the TiN film.

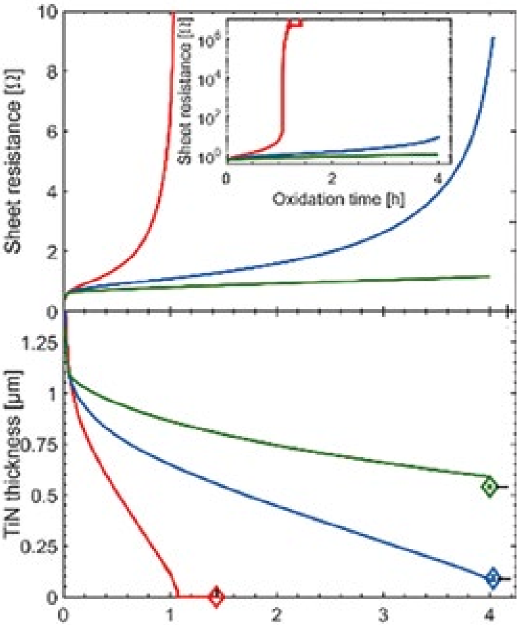


Fig. 1: *Top: in situ measured sheet resistance of TiN thin films over oxidation time for three samples oxidized at 600, 650, and 700 °C; Bottom: remaining TiN thicknesses depending on oxidation time calculated from measured sheet resistances. Diamonds indicate remaining TiN film thickness measured ex situ by STEM.*

In summary, it was shown that oxidation behaviour [2] and phase transitions [1] can be tracked by *in situ* sheet resistance measurements making it an autonomous self-reporting property for monitoring the residual lifetime of components during application. These proof of concept demonstrations have fuelled the implementation of spatially resolved resistivity measurements in the "Thin Films & Nanostructured Materials group" of the SN department and also serve as one thin film materials design criterion now explored within the newly setup of the MPG group "Thin Film Materials Design" (see p. 26).

References

1. B. Stelzer, X. Chen, P. Bliem, M. Hans, B. Volker, R. Sahu, C. Scheu, D. Primetzhofer, J.M. Schneider: Sci. Rep.-UK 9 (2019) 7.
2. B. Stelzer, M. Momma, J.M. Schneider: Adv. Funct. Mater. (2020) 2000146.

Max Planck fellow group: Electrochemistry & Nanoscale Materials

Group head: K. Tschulik

Faculty of Chemistry and Biochemistry, Analytical Chemistry II, Ruhr-Universität Bochum

The development of improved electrocatalysts for renewable energy technologies and the synthesis of industrially important chemicals are hampered by the current lack of information on favourable catalyst properties. The aim of this Max Planck Fellow group is to understand the influence of compositional and structural properties of materials on their electrocatalytic activity, to tackle this problem and pave the way to reaction-specific, rational catalyst design by dissecting the most relevant criteria. For this purpose, we will systematically alter the relevant materials properties, including composition, grain size, surface facets, and porosity and examine their electrocatalytic performance. We will follow a concerted interdisciplinary approach to gain better insights into the underlying processes. We seek to establish innovative synthesis and processing steps for catalyst materials, such as alloy electrodeposition and magnetic field-assisted electrochemical post-processing. The obtained materials will be utilized as catalysts with systematically tuned and precisely characterized properties to identify their performance in terms of electrocatalytic activity, selectivity and durability.

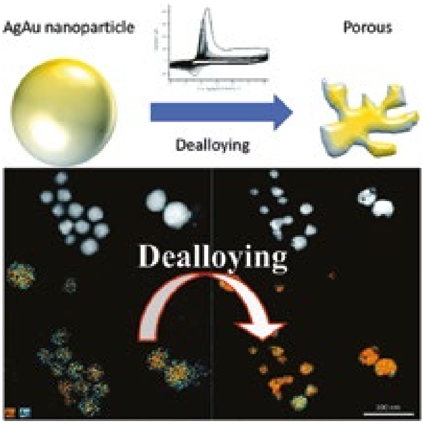


Fig. 1: *Schematic representation of nanoparticle modification by dealloying, cyclic voltammetry data and identical location STEM images and EDX mapping, before and after dealloying of AgAu nanoparticles [1].*

A first impression on the great potential of nanoparticle post-processing by electrochemical dealloying combined with identical location transmission electron microscopy (TEM) (Fig. 1) has been obtained for conventionally-synthesized AuAg nanoparticles in a collaboration with the Scheu group [1, see p. 13].

In addition to established methods for electrochemical characterization of large numbers of nanoparticles immobilized on a surface, we will use single nanoparticle electrochemistry to characterize reactions at or of individual nanoparticles, allowing us to determine the intrinsic electrochemical response of nanomaterials one by one (Fig. 2). For instance, by performing electrocatalytic single entity studies, we have demonstrated that the oxygen evolution reaction is limited by removal of the product from individual $CoFe_2O_4$ nanocatalysts in alkaline media and at current densities of several kA/m^2 [2].

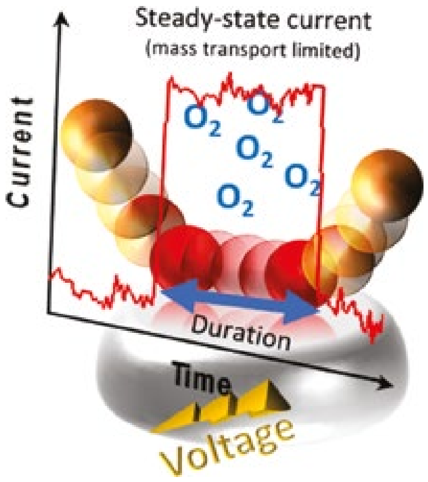


Fig. 2: *Scheme of single particle electrocatalysis at individual NPs.*

Furthermore, in depth *ex situ* characterization and operando spectro-electrochemistry will be used to account for changes in properties, activities and selectivity during electrocatalysis. These experimental tasks will be supported by state-of-the-art computational studies conducted at the MPIE, to gain fundamental insights into catalyst restructuring under operando conditions and into the atomic-scale processes occurring at the solid/liquid interface.

References

1. Rurainsky, C.; Garzón Manjón A.; Hiege, F.; Chen, Y.T.; Scheu, C.; Tschulik, K.: J. Mater. Chem. A 8 (2020) 19405.
2. El Arrassi, A.; Liu, Z.; Evers, MV.; Blanc, N.; Bendt, G.; Saddeler, S.; Tetzlaff, D.; Pohl, D.; Damm. C.; Schulz. S.; Tschulik. K.: J. Am. Chem. Soc. 141 (2019) 9197.

MPG group: High temperature materials

Group head: G. Eggeler

Chair for Materials Science and Engineering, Ruhr-Universität Bochum

High temperature materials have always been one of the research strong holds of the MPIE. In 2010, a Max Planck Fellow High Temperature Materials (HTM) group led by G. Eggeler was established in this area, which continued seamlessly as an external MPIE research group since June 2016. HTM establishes an operational research link between the Chair for Materials Science at the Ruhr-Universität Bochum (RUB) and the MPIE. From the beginning on, A. Kostka was closely associated with the HTM research. His present permanent position as a transmission electron microscopy (TEM) scientist at the RUB allows him to participate in HTM research and to provide scientific contributions. HTM research topics focus on material properties, which strongly depend on microstructural evolution during high temperature exposure. One objective of HTM is to improve advanced structural materials based on an in depth understanding of elementary deformation and damage processes, which govern high temperature strength. But topics like high temperature shape memory alloys also receive attention.

Ni-base single crystal super alloys: In the case of Ni-base single crystal super alloys, which can resist mechanical loads at temperatures well above 1000 °C, there is an interest in the segregation tendencies of Re, which is known to significantly improve creep strength. HTM efforts concentrated on segregation phenomena of Re at low angle grain boundaries (in cast microstructures) [1] and at γ/γ’-interfaces during creep [2, 3].

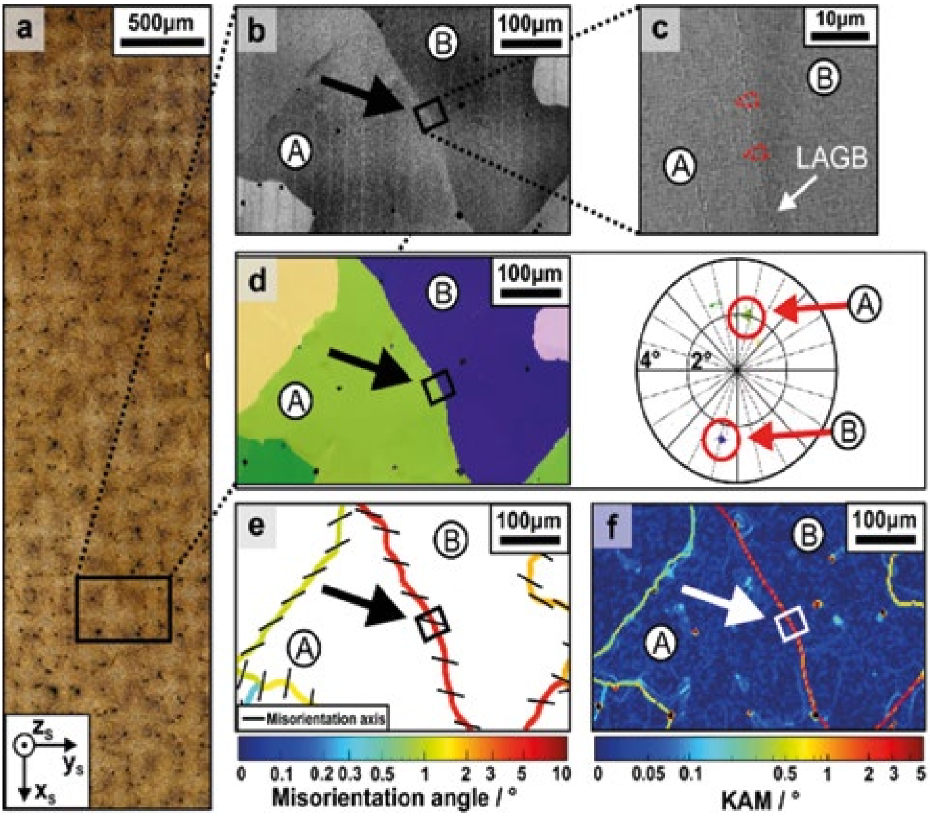


Fig. 1: *Identifying locations for subsequent APT analysis [1]. (a) Optical micrograph of dendritic microstructure. (b) Backscatter electron) (BSE) overview image. (c) Higher magnification micrographs showing a low angle grain boundary between the regions marked with A and B. APT specimens containing this boundary were subsequently extracted [1]. (d) Orientation of regions A and B. (e) Corresponding misorientation angles. (f) KAM map.*

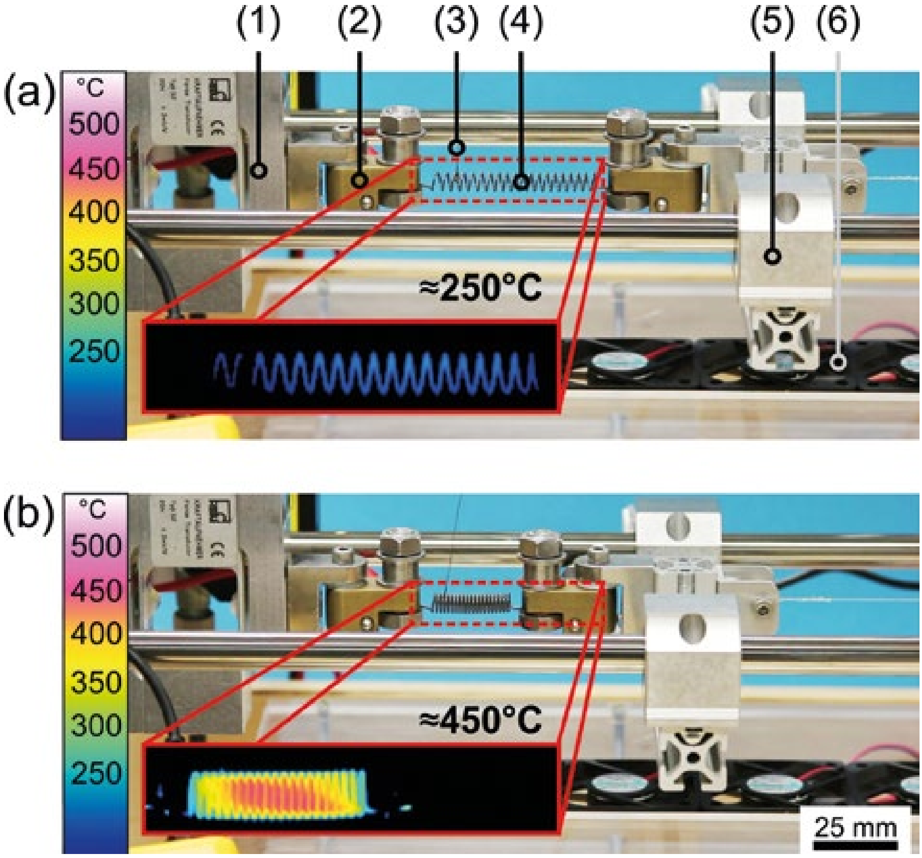


Fig. 2: *Test rig for assessing Ti-Ta HTSMA spring actuator performance [6]. The two photographs were taken during a heating cycle. The insets show colour coded temperature distributions recorded with an IR thermo camera. Key elements of the test rig are highlighted in a) 1 – load cell; 2 – grips (for mechanical clamping)/ electrical contacts (for direct current heating); 3 – control thermo couple; 4 – Ti-Ta spring; 5 – low friction linear slide system; 6 – fan array (for air cooling). (a) Begin of heating cycle – martensitic state, T ≈ 250 °C. (b) Reaching end of heating cycle – directly after reverse transformation to austenite, T ≈ 450 °C.*

As an example, Fig. 1 shows the detailed characterization of an as-cast microstructure by means of optical microscopy and electron backscatter diffraction (EBSD) in the scanning electron microscope (SEM). Specific locations were identified, and, in a second step, these regions were chemically analyzed using atom probe tomography (APT) in B. Gault's group (results not shown here). The super alloy research was integrated into the research of the collaborative research center SFB/TR 103 [4, see p. 87] funded by the German Research Foundation (DFG), where MPIE is a partner. From Bochum P. Thome, F. Scholz and J. Frenzel actively contributed to HTM research.

High temperature shape memory alloys: The HTM group also works on functional materials. Shape memory alloys (SMAs) can recover their original shape after severe plastic deformation. The underlying one way effect relies on a diffusionless phase transformation, where a low temperature phase (detwinned martensite) retransforms into a high temperature phase austenite. Conventional SMAs show transformation temperatures in the 50 °C temperature range. There is a strong interest in SMA systems, which can operate at higher temperatures. From a fundamental point of view, this is challenging, because diffusional processes start to interfere with the physical nature of the martensitic transformation. In the reporting period, the HTM group performed theoretical and experimental research on the TiTa system, in a DFG funded research activity (FOR 1766). In collaboration with the Interdisciplinary Centre for Advanced Materials Simulation (see p. 79) it was shown that small additions of Sc can suppress the detrimental formation of the ω-phase, which makes it difficult to apply binary TiTa high temperature shape memory alloys as actuators [5]. It was experimentally demonstrated that TiTa spring actuators can be processed and that the one way effect can be exploited at temperatures above 400 °C [6], Fig. 2.

References

1. He, J.Y.; Scholz, F.; Horst, O.M.; Thome, P.; Frenzel, J.; Eggeler, G.; Gault, B.: Scripta Mater. 185 (2020) 88 and 187 (2020) 309.
2. He, J. Y.; Cao, L.J.; Makineni, S.K.; Gault, B.; Eggeler, G.: Scripta Mater. 191 (2021) 23.
3. Wu, X.X; Makineni, S.K.; Kontis, P.; Dehm, G.; Raabe, D.; Gault, B.; Eggeler, G.: Materialia 4 (2018) 109.
4. https:/www.sfb-tr 103.de
5. Ferrari, A.; Paulsen, A.; Langenkämper, D.; Piorunek, D.; Somsen, C.; Frenzel, J.; Rogal, J.; Eggeler, G.; Drautz, R.: Phys. Rev. Mat. 3 (2019) 103605.
6. Paulsen, A.; Dumlu, H.; Piorunek, D.; Langenkämper D.; Frenzel, J.; Eggeler, G.: Shape Mem. Superelastictiy 7 (2021) 222.

MPG group: De magnete – Designing magnetism on the atomic scale

Group head: O. Gutfleisch

Functional Materials, TU Darmstadt

Hard and soft magnets are everywhere in our modern society. They are key components in advanced energy conversion and energy recovery systems, electromobility and transport systems, data storage and communication, consumer electronics, magnetic resonance imaging and magnetic separation, robotics and automation, electric power transmission and distribution, and possibly also in new magnetic refrigeration technologies.

Based on this, we established in autumn 2020 the external research group *De Magnete* with the aim to push functional bulk magnets to their physical limits given by their intrinsic properties. Key is the understanding of the critical magnetization reversal processes on the atomic scale.

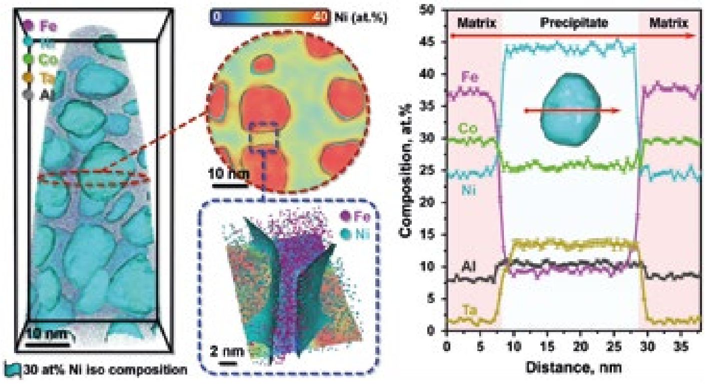


Fig. 1: *Ultrastrong and ductile bulk high-entropy alloys with excellent soft magnetic properties were achieved by introducing a high volume fraction of coherent and ordered nanoprecipitates into the alloy matrix. The high lattice coherency between the homogeneously dispersed nanoprecipitates and the alloy matrix creates only negligible distortion fields on magnetic domain wall motion and contributes significantly to the strength.*

Transitional and dynamic processes are relevant for nucleation and propagation during all critical magnetization reversal processes. It is one of the great challenges in magnetism and magnetic materials for advanced characterization and simulation to capture, reproduce and finally predict these phenomena across all spatial and temporal scales. There is the need for overcoming the disconnection of the modelling methodologies and also for validating atomistic and thus highly selective experimental observations on the meso- and macroscales.

We are searching for the perfect defect; an ideal perfect single crystal will allow us to determine unambiguously the physical limit of e.g. a magnetic material based on its intrinsic properties (saturation magnetization, magnetocrystalline anisotropy field and Curie temperature and derived from these, their characteristic micromagnetic length scales).

We want to explore and master the duality of defects – they can alter local magnetic anisotropy and with this serve as a nucleation site or act as a pinning site. We can observe local softening detrimental for a permanent magnet or we can imprint a useful step or gradient in K, the anisotropy constant. We want to understand the electrostatic potential, size, symmetry, coherency, orientation, interfacial sharpness, population density, scale, embedment in the surrounding matrix of the defect and its thermodynamic stability.

Based on this understanding, we will develop a theory driven approach for alloy and microstructure design, as well as defect (of various dimensionality) manipulation of hard, soft, magnetocaloric materials and extend this to new concepts like high entropy alloys and additive manufacturing, where we then look into the superior mechanical properties and new geometries.

Finally, we are running the biweekly *De Magnete* seminar series between the two groups at TU Darmstadt and MPIE Düsseldorf to cross-fertilize the interaction.

References

1. L. Han, Z. Rao, I.R. Souza Filho, F. Maccari, Y. Wei, G. Wu, A. Ahmadian, X. Zhou, O. Gutfleisch, D. Ponge, D. Raabe, Z. Li: Adv. Mater. 33 (2021) 2102139 .
2. S. Ener, K.P. Skokov, D. Palanisamy, T. Devillers, J. Fischbacher, G. Gomez Eslava, F. Maccari, L. Schäfer, L.V.B. Diop, I. Radulov, B. Gault, G. Hrkac, N.M. Dempsey, T. Schrefl, D. Raabe, O. Gutfleisch: Acta Mater. 214 (2021) 116968.
3. X. Ye, F. Yan, L. Schaefer, D. Wang, H. Geßwein, W. Wang, M.R. Chellali, L.T. Stephenson, K. Skokov, O. Gutfleisch, D. Raabe, H. Hahn, B. Gault, R. Kruk: Adv. Mater. (2020) 2006853.
4. A.M. Schönhöbel, R. Madugundo, J.M. Barandiarán, G.C. Hadjipanayis, D. Palanisamy, T. Schwarz, B. Gault, D. Raabe, K. Skokov, O. Gutfleisch, J. Fischbacher, T. Schrefl: , Acta Mater. 200 (2020) 652.
5. D. Palanisamy, S. Ener, F. Maccari, L. Schäfer, K.P. Skokov, O. Gutfleisch, D. Raabe, B. Gault: Phys. Rev. Mater. 4 (2020) 054404.
6. Z. Rao, B. Dutta, F. Körmann, D. Ponge, L. Li, J. He, L. Stephenson, B. Gault, L. Schäfer, K.P. Skokov, O. Gutfleisch, D. Raabe, Z. Li: Phys. Rev. Mater. 4 (2020) 014402.

MPG group: Thin film materials design

Group head: J. M. Schneider

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Independent research group continuing MP fellowship, start 01 October 2020

Thermal stability of protective coatings is one of the performance-defining properties for advanced cutting and forming applications as well as during energy conversion. To investigate the effect of oxygen incorporation on the high-temperature behaviour of (Ti,Al)N, metastable cubic (Ti,Al)N and (Ti,Al)(O_xN_{1-x}) coatings were synthesized using reactive arc evaporation and the results were published in Acta Materialia [1]. X-ray diffraction of (Ti,Al)N and (Ti,Al)(O_xN_{1-x}) coatings revealed that spinodal decomposition is initiated at approximately 800 °C, while the subsequent formation of wurtzite solid solution was clearly delayed from 1000 °C to 1300 °C for (Ti,Al)(O_xN_{1-x}) compared to (Ti,Al)N (Fig. 1). This thermal stability enhancement can be rationalized based on calculated vacancy formation energies in combination with spatially-resolved composition analysis and calorimetric data: Energy dispersive X-ray spectroscopy and atom probe tomography data indicated a lower O solubility in wurtzite solid solution compared to cubic (Ti,Al)(O,N).

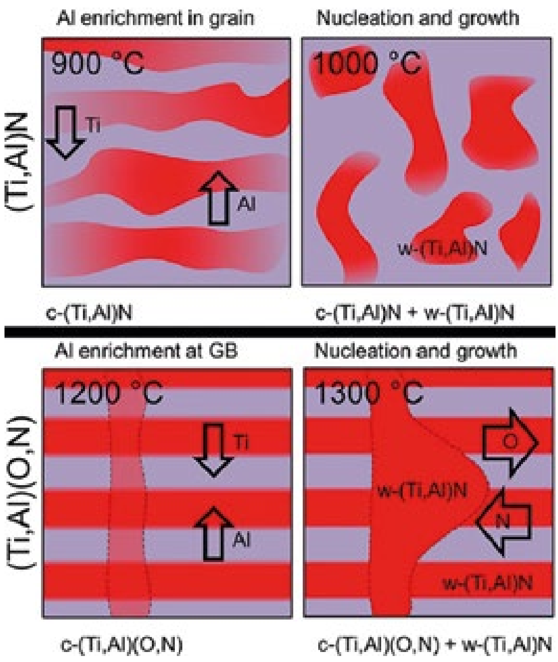

Fig. 1: *Schematic representation of decomposition of rotated c-(Ti,Al)N and c-(Ti,Al)(O,N) coatings; adapted from [1].*

Hence, it is evident that for the growth of the wurtzite, AlN-rich phase in (Ti,Al)N, only mobility of Ti and Al is required, while for (Ti,Al)(O,N), in addition to mobile metal atoms, also non-metal mobility is required. Prerequisite for mobility on the non-metal sublattice is the formation of non-metal vacancies, which require larger temperatures than for the metal sublattice due to significantly larger magnitudes of formation energies for the non-metal vacancies compared to the metal vacancies. This notion is consistent with calorimetry data, which indicate that the combined energy necessary to form and grow the wurtzite phase is larger by a factor of approximately two in (Ti,Al)(O,N) than in (Ti,Al)N, causing the here reported thermal stability increase.

References

1. D. Holzapfel, D. Music, M. Hans, S. Wolff-Goodrich, D. Holec, D. Bogdanovski, M. Arndt, A. O. Eriksson, K. Yalamanchilif, D. Primetzhofer, C. H. Liebscher, J. M. Schneider: Acta Mater (2021) 117204.

Additional selected publications

2. B. Völker, C. Du, H. Fager, H. Rueß, R. Soler, C. Kirchlechner, G. Dehm, J.M. Schneider: Surf. Coat. Tech. 390 (2020) 125645.
3. B. Völker, B. Stelzer, S. Mráz, H. Rueß, R. Sahu, C. Kirchlechner, G. Dehm, J.M. Schneider: Mater. Design 206 (2021) 109757.
4. D. Göhl, H. Rueß, S. Schlicht, A. Vogel, M. Rohwerder, K.J.J. Mayrhofer, J. Bachmann, Y. Román-Leshkov, J.M. Schneider, M. Ledendecker: Chem. Electro. Chem. 7 (2020) 2404.
5. D. Göhl, H. Rueß, M. Pander, A.R. Zeradjanin, K.J.J. Mayrhofer, J.M. Schneider, A. Erbe, M. Ledendecker: J. Electrochem. Soc. 167 (2020) 021501.
6. S. Evertz, I. Kirchlechner, R. Soler, C. Kirchlechner, P. Kontis, J. Bednarcik, B. Gault, G. Dehm, D. Raabe, J.M. Schneider: Mater. Design 186 (2020) 9.
7. S. Evertz, S. Prünte, L. Patterer, A. Marshal, D.M. Holzapfel, A. Schökel, M. Hans, D. Primetzhofer, J.M. Schneider: Cond. Mat. 5 (2020) 18.
8. S. Evertz, V. Schnabel, M. Köhler, I. Kirchlechner, P. Kontis, Y.-T. Chen, R. Soler, B.N. Jaya, C. Kirchlechner, D. Music, B. Gault, J.M. Schneider, D. Raabe, G. Dehm: Front. Mater. 7 (2020).
9. S. Evertz, J.M. Schneider: Materials 13 (2020) 4911.
10. A. Marshal, P. Singh, D. Music, S. Wolff-Goodrich, S. Evertz, A. Schökel, D.D. Johnson, G. Dehm, C.H. Liebscher, J.M. Schneider: J. Alloy. Compd. 854 (2021) 155178.
11. S. Wolff-Goodrich, A. Marshal, K.G. Pradeep, G. Dehm, J.M. Schneider, C.H. Liebscher: J. Alloy. Compd. 853 (2021) 156111.

Interdepartmental group: Sustainable synthesis of materials

Group heads: I. R. Souza Filho, Y. Ma

MA

The high demand for metallic materials in key sectors such as energy, construction, safety, and transport results in predicted production growth rates of metals up to 200 % until 2050. Yet, most of the metallic materials, particularly steel, aluminium, nickel, and titanium, require a lot of energy when they are extracted and manufactured from ores. This fact means that metallic industries have become a huge environmental burden, with a total energy consumption of ~53 EJ (10^{18} J, that is 9 % of the global energy used) and almost 35 % of industrial CO_2-equivalent emissions (4.4 Gt) when counting only steel and aluminium industries [1, 2].

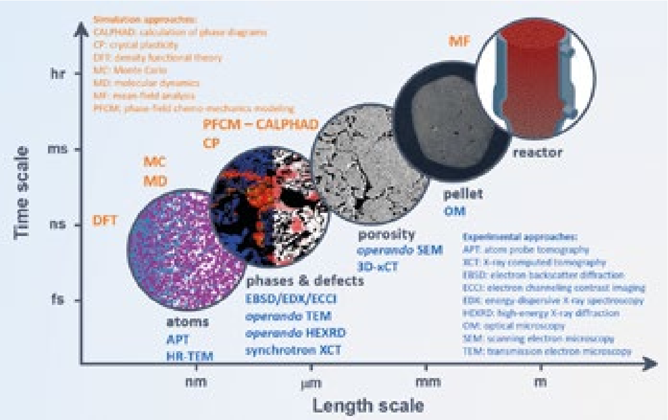

Fig. 2: *Multiscale approaches towards better understanding of the materials' synthesis processes.*

The interdepartmental group "Sustainable Synthesis of Materials" focuses on the physical and chemical foundations to improve the direct sustainability (related to synthesis and manufacturing) of structural and functional metals. The research areas include reduced CO_2-intense production, low-energy material synthesis, recycling, scrap-compatible alloy design, sustainable semiconductors, improved longevity of alloys, and green energy generation.

The ongoing research activities lie in the exploitation of sustainable routes for iron production using various hydrogen carriers (*e.g.,* pure H_2 gas or mixtures, NH_3, and hydrogen plasma, *etc.*) as reductants as well as via low-temperature electrolysis (*i.e.,* up to 200 °C) (Fig. 1). The hydrogen-based reduction methods include solid-state direct reduction with molecular H_2 [3], hydrogen plasma-based reduction of molten ores [4] as well as a hybrid method combining both. The group also investigates the impact of microstructural aspects of iron powders on their combustion for green energy generation (Fig. 1**)**. The focus of those studies is placed on the reaction thermodynamics, kinetics, associated phase transformations, microstructural evolutions, and transport phenomena across several length and time scales, down to the atomistic and electronic mechanicsms associated with these redox reactions (Fig. 2**)**. Particularly, multiscale and *operando* experimental methods are employed to reveal the underlying reaction mechanisms in these synthesis processes. Also, physics-based numerical modelling is applied to interpret experimental findings and aid the design of key experiments in collaboration with in-house theory groups.

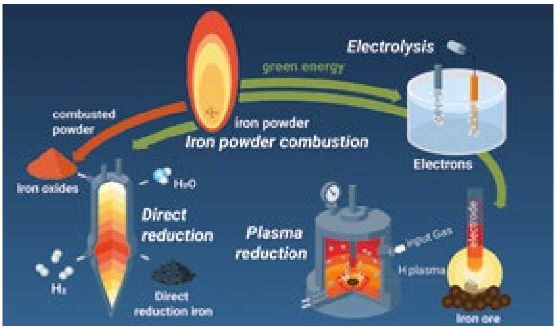

Fig. 1: *The group focuses on the chemical and physical foundations for sustainable synthesis of iron and green energy generation.*

The interdepartmental group "Sustainable Synthesis of Materials" evolved from the interdepartmental group "Physical Metallurgy of Sustainable Metals", which was led by D. Ponge and D. Raabe till September 2021, to match the need for interdisciplinary cooperation between the departments.

References

1. Allwood, J.M.; Cullen, J.M.; Carruth, M.A.; Cooper, D.R.; McBrien, M.; Milford, R.L.; Moynihan, M.C.; Patel, A.C.H.; Bauer, S.: Sustainable materials: With both eyes open. UIT Cambridge Ltd (2012).
2. Raabe, D.; Tasan, C.C.; Olivetti E.A.: Nature 575 (2019) 64.
3. Kim S.H.; Zhang X.; Ma Y.; Souza Filho I.R.; Schweinar K.; Angenendt K.; Vogel D.; Stephenson L.T.; El-Zoka A.A.; Mianroodi J.R.; Rohwerder M.; Gault B.; Raabe D.: Acta Mater. 212 (2021) 116933.
4. Souza Filho I.R.; Ma Y.; Kulse M.; Ponge D.; Gault B.; Springer H.; Raabe D.: Acta Mater. 213 (2021) 116971.

Interdepartmental group: Materials science of additive manufacturing

Group heads: R. Ramachandramoorthy[1], H. Springer[2]

[1] SN, [2] MA, now also at: Institute of Metal Forming, RWTH Aachen University, Germany

The new group of “Materials science of additive manufacturing” headed by R. Ramachandramoorthy and H. Springer was established in 2021, to leverage the selective laser melting (SLM) technique as a unique multifaceted tool for novel materials design, bundeling the strong research links between the departments in this field.

A plethora of materials characterization efforts have been undertaken over the years to understand the influence of processing parameters on the microstructure of the fabricated parts using SLM. But a concerted effort exploiting simultaneously the material and structural effects to achieve mechanical and physical performance enhancement is largely missing in the field of SLM and this forms the core motivation for this group. One of the key research foci for the group will be tailored alloy development towards achieving specific microstructure and material properties. This aim will be approached using a combination of customized powder development and state-of-the-art alloy design concepts. Specifically, a laboratory-scale powder development system will be implemented to obtain small quantities of SLM powders from pre-selected and custom cast alloys. A few material design strategies currently envisioned include stiff-and-light high modulus steels for lightweight applications (Fig. 1), magnetic and fuel cell materials, as well as metal-matrix composites produced by reactive atmosphere based SLM [2].

The second research focus of the group will be on the optimization of microstructure and site-specific laser processing parameters to tune the mechanical properties of 3D topologies such as complex truss-based lattices, plate-based lattices and lattices with doubly curved geometries based on triply periodic minimal surfaces (Fig. 2). Specifically, the rate-dependent (0.001/s to 1000/s) microstructure and structural evolution of the different architectures will be investigated using a combination of experimental mechanical metrology and finite element method-based simulations. Particular attention will be on minimizing the detrimental effects of surface sintering and lattice defects on the mechanical properties using a combination of post-processing techniques such as heat-treatments, chemical etching and sand-blasting and evaluated via MicroCT analysis. The ultimate goal is to establish microstructure-architecture-mechanical property relationships in light-weight low density lattices.

References

1. Springer, H.; Baron, C.; Mostaghimi, F.; Poveleit, J.; Mädler, L.; Uhlenwinkel, V.: Addit. Manuf. 32 (2020) 101033.
2. Springer, H.; Baron, C.; Szczepaniak, A; Jägle, E.A.; Wilms, M.B.; Weisheit, A.; Raabe, D.: Mater. Des. 111 (2016) 60.

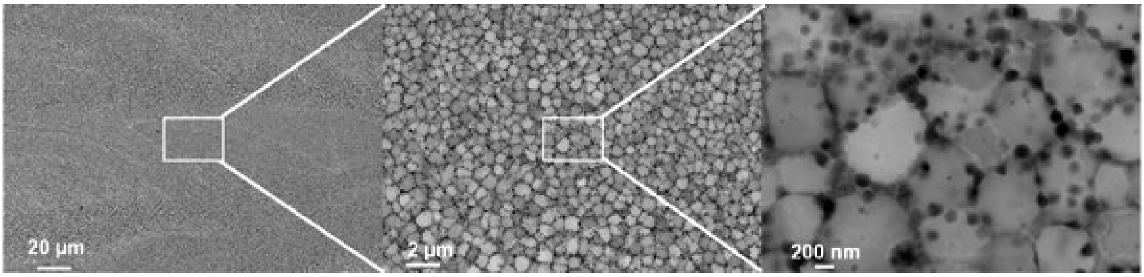

Fig. 1*: Novel materials through novel synthesis: the in situ nanostructuring of* TiB_2 *particles in a ferritic matrix during SLM of an FeTiB alloys ensures a unique property combination of high stiffness and strength coupled with low density [1].*

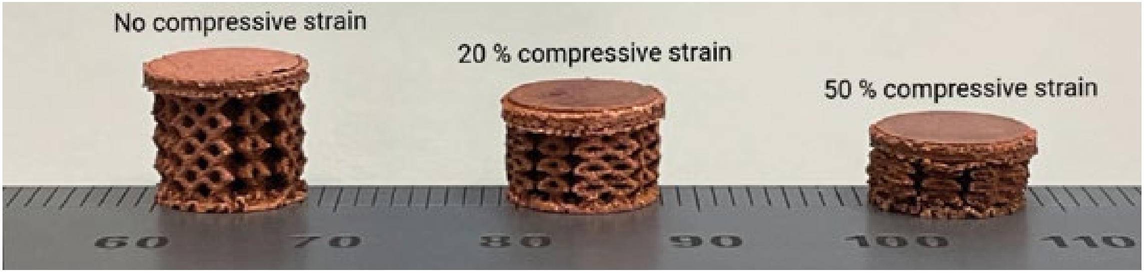

Fig. 2: *Millimeter scale pure Cu octet lattices in the undeformed and after quasi-static deformation at different strains.*

Interdepartmental group: Sustainable material science and technology

Group head: H. Springer

MA, now also at: Institute of Metal Forming, RWTH Aachen University, Germany

The RWTH Aachen University partner group on “Sustainable Material Science and Technology” investigates and develops innovative strategies for the sustainable production of metallic materials. We aim at a holistic approach to reduce the environmental impact, by integrating aspects of synthesis science and technology, such as energy consumption and emission of climate gases, into the material design, i.e. the use of alloying elements, tolerance for impurities and optimisation of the property profile for lightweight constructions. Associated research topics span from combinatorial alloy design of Mg-Al-Ca composites, development of high stiffness and self-healing materials, fundamentals of recrystallisation during thermo-mechanical treatments of composite materials, to the development of damage tolerant dual phase steels. This is done in the framework of two collaborative research centers as well as in projects funded by the AiF (Arbeitsgemeinschaft industrieller Forschungsvereinigungen) and BMWi (Federal Ministry for Economic Affairs and Energy), all being integrated into the activities of the academic and research department for metallic composite materials at the RWTH Aachen University.

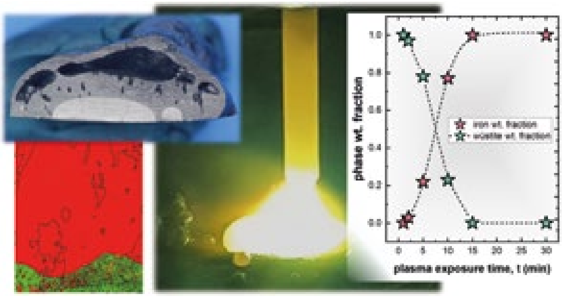

Fig. 1: *Investigating the hydrogen plasma smelting reduction of iron ores at the MPIE: From model processes to microstructure characterisation and evaluation of the reduction kinetics.*

The focus of our activities on sustainable metallurgy lies on investigating novel concepts for the reduction of iron ores. The transformation of the steel industry from coal-based blast furnace technology towards the final goal of sustainable hydrogen metallurgy is a key pathway to reduce the global carbon emissions, but represents a multitude of scientific and technological challenges, the solution of which requires input from all departments. Two processing routes are being investigated: The direct reduction process, where ore particles or pellets are transformed to iron in a hydrogen containing atmosphere, is from a technological standpoint already on a high readiness level, but the underlying microstructural, transport and transformation phenomena are not yet fully understood. We aim to elucidate these mechanisms with advanced microstructure characterisation techniques, small-scale test reactor set-ups, simulations down to electronic scales and advanced *in situ* electron microscopy, thus bundeling core competences from all departments [1]. This does not only provide the basis for further optimisation of the reduction process, but also transfers into other upcoming technological innovations such as the use of metallic powders as a sustainable and climate neutral fuel.

The other relevant process is the hydrogen plasma smelting reduction, where the ore is molten and simultaneously reduced in an electrical arc furnace with a hydrogen plasma arc. This process offers the greatest ecological and economical potential, as it offers thermodynamic advantages by eliminating the need to subsequently melt the reduced iron, as well as much accelerated reduction kinetics due to the stronger reduction potential and energy level of ionised hydrogen species (Fig. 1). However, while it is expected that existing electric arc furnaces can be transformed into hydrogen plasma reactors, a multitude of scientific questions and engineering challenges have to be addressed in order to bring this most promising hydrogen metallurgy technique onto a higher technology readiness level. These involve especially the nature of the interaction between the high energy plasma and ionised liquid oxides, the role of alloying elements on the reduction chemistry and slag formation, as well as gas composition, processing stability and furnace construction. All these topics have to be optimised simultaneously to ensure an optimised hydrogen efficiency as being cost effective and technologically feasible in view of the massive scale of global steel production. In order to efficiently elucidate and address the vast range of fundamental research questions, we develop and deploy targeted experimental setups – for example lab scale direct reduction cells [2] and plasma reactors [3] together with modelling approaches in close cooperation with other groups at the MPIE. Currently the focus lies on the incorporation of *in situ* diagnostics for example by mass spectroscopy analysis of the reduction products close to the reaction zone, as well as novel strategies to plasma electrode design. The transferability of the derived results towards the industrial scale is discussed with external partners.

References

1. Kim, S.-H.; Zhang, X.; Schweinar, K.; Souza Filho, I.; Angenendt, K.; Ma, Y.; Vogel, D.; Stephenson, L.; El-Zoka, A.; Mianroodi, J.; Rohwerder, M.; Gault, B.; Raabe, D.: Acta Mater. 212 (2021) 116933.
2. Auinger, M.; Vogel, D.; Vogel, A.; Spiegel, M.; Rohwerder, M.: Rev. Sci. Instrum. 84 (2013) 085108.
3. Souza Filho, I.R.; Ma., Y.; Kulse, M.; Ponge, D.; Gault., B.; Springer, H.; Raabe, D.: Acta Mater. 213 (2021) 11697.

Interdepartmental group: Hydrogen embrittlement in high-performance alloys

Group head: B. Sun

MA

Hydrogen (H), the most abundant element in the universe, offers an attractive sustainable energy solution that can substitute carbon-based fuels and thus reduce carbon dioxide (CO_2) emissions. The infrastructures and transport means required for the implementation of a H economy require affordable and mass-produced high-strength and sustainable materials. However, when the small and light H atoms make the way into a high-strength alloy (strength above ~650 MPa), the material's load-bearing capacity is abruptly lost, a phenomenon known as H embrittlement [1, 2]. This embrittlement effect often occurs in an unpredictable manner, as the ingress of the ubiquitous H into a material is difficult to avoid and a H concentration of only a few parts per million by weight is often sufficient to cause catastrophic failure [1, 3, 4]. Thus, H embrittlement can basically threaten any industrial and societal applications in a H economy that aim to use high-performance alloys to make structural components.

The group was established in 2021, and aims to understand the mechanisms of H embrittlement, as well as to use the acquired knowledge to design novel microstructure concepts with enhanced H-resistance. Ultimately, we aim to promote the development of high-performance and H-tolerant alloys that are urgently needed for the dawn of the H age.

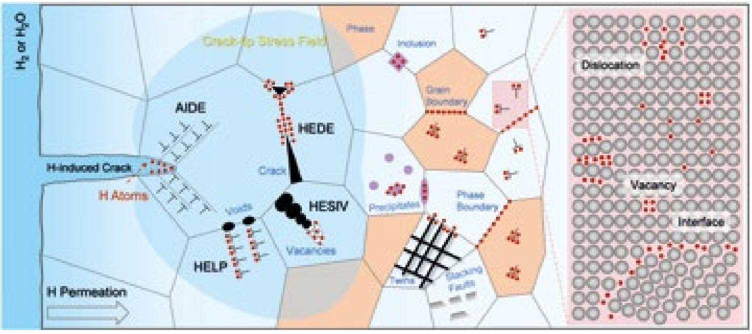

Fig. 1: *Schematic diagram showing the behaviour of H trapping, H-defect interactions and micromechanical models for the formation of H-induced damage in metallic materials.*

The ongoing research activities include exploring three fundamental aspects of H embrittlement: H trapping and diffusion, H-defect interactions and H-induced damage evolution. Special focuses are placed on their interplay, the association with the activation of different micromechanical models (e.g. H-enhanced decohesion (HEDE), H-enhanced localized plasticity (HELP), adsorption-induced dislocation emission (AIDE), and H-enhanced strain-induced vacancies (HESIV)), and their essential influences on the H-induced degradation of mechanical properties (Fig. 1). The research is carried out by state-of-the-art characterization techniques down to the atomic level. Multi-scale simulation methods are applied in collaboration with simulation groups at the MPIE, both at the electronic and continuum scale.

Besides studying H embrittlement mechanisms, the group also performs thermodynamics-based microstructural design in order to enhance alloys' H-resistance at a minimum cost of their mechanical performance. Our current strategies include producing theory-guided H-trapping sites to suppress H diffusion and/or forming tough microstructural ingredients to suppress H-induced crack nucleation, growth, and percolation. As an example, we have exploited the typically undesired chemical heterogeneity within the material's microstructure that enables local enhancement of crack resistance and local H trapping. We used this approach in a manganese-containing high-strength steel and produced a high dispersion of manganese-rich zones within the microstructure. These solute-rich buffer regions allowed for local micro-tuning of the phase stability, arresting H-induced microcracks and thus resulting in a superior H embrittlement resistance (better by a factor of two) without sacrificing the material's strength and ductility [1].

This group is a good example of the intense interdepartmental collaboration: understanding of trapping sites for hydrogen and the effect of hydrogen on interfacial decohesion come from *in situ* calculations; materials reponse under the influence of hydrogen is probed under environmental and *in situ* microscopy conditions; spatially resolved observation of hydrogen is enabled by Kelvin probe and atom probe analysis; and advanced microstructure concepts for making materials more resistant to hydrogen come from alloy design and processing.

References

1. Sun, B.; Lu, W.; Gault, B.; Ding, R.; Makineni, S. K.; Wan, D.; Wu, C.-H.; Chen, H.; Ponge, D.; Raabe, D.: Nat. Mater. (2021) https://doi.org/10.1038/s41563-021-01050-y.
2. Raabe, D.; Tasan, C. C.; Olivetti, E. A.: Nature 575 (2019) 64.
3. Breen, A. J.; Stephenson, L.; Sun, B.; Li, Y.; Kasian, O.; Raabe, D.; Herbig, M.; Gault, B.: Acta Mater. 188 (2020) 108.
4. Sun, B.; Krieger, W.; Rohwerder, M.; Ponge, D.; Raabe, D.: Acta Mater. 183 (2020) 313.

Max Planck partner group: Combinatorial design of novel high-performance permanent magnets

Group head: P.K. Gokuldoss

Indian Institute of Technology, Madras, India

The persistent increase in prices of rare-earth (RE) elements such as Nd and Dy, along with the low operating temperature (100-200 °C) of (Nd, Dy)-Fe-B magnets has led to the search for new RE lean permanent magnetic alloys. Typically, Sm-Co based and Nd-Fe-B based permanent magnets are widely used in automobiles, energy conversion systems, power generation etc. With the advent of electric vehicles and advanced mobility systems for mass transportation in addition to increased emphasis on clean energy, the demand of high-performance permanent magnets which are either RE free or lean-RE compositions is significantly higher. The objective of this partner group is to design and develop novel high-performance magnets using combinatorial methods that are lean in RE concentrations.

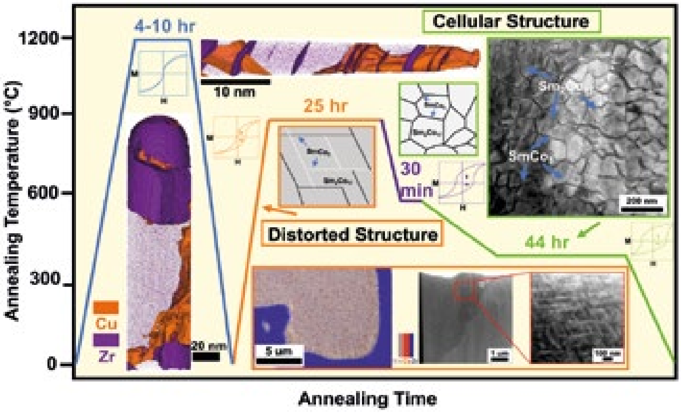

Fig. 1: *Thermomagnetic (Temperature vs time) sequence plot indicating the evolution of nanostructure in a Sm-Co based high-performance permanent magnet. The inset shows elemental distribution maps with Zr and Cu delineated using iso-concentration surfaces highlight the difference between well-formed cellular structures exhibiting high coercivity state and those that are partially formed (elongated grains in transmission electron microscopy inset image with blocky Zr segregations) or having decomposed states with low coercivity values not suitable for permanent magnetic applications.*

To address the above objective, the concept of multi-component alloy design is utilized wherein novel compositions are explored for identification of unique phases that can exhibit spontaneous magnetization. The use of combinatorial approaches including reaction sintering and rapid annealing enables exploring and understanding the role of alloying elements (e.g. Fe, Cu, Zr) and their content on phase formation, microstructure evolution and magnetic properties especially for Sm-Co based magnets [1].

More specific, the development of Sm_2Co_{17} cell structures with the Cu-rich cell boundary phase intertwined by the Zr platelets are ideal configurations, which exhibit high coercivity values as shown in Fig. 1. It implies that the sequence of thermo-magnetic treatments required for the partitioning of constituent elements such as Cu and Zr into their platelet forms is currently being developed with particular focus on the fundamental diffusion aspects. Especially the role of annealing temperature and time on chemical separation, followed by precipitation and growth of Cu and Zr enriched phases from their initial solid solution state, are current matters of deeper investigation. Such understanding of elemental segregation effects at nano-meter scale during processing is crucial towards developing robust thermomagnetic procedures for the development of novel high-performance permanent magnets.

In addition, novel multi-component low-melting (eutectic) phases are also being designed to be used as grain boundary 'infiltrators' which can separate neighbouring magnetic domains and hence enhance coercivity. To this end, the concept of grain boundary engineering is utilized to understand the grain boundary characteristics of the sintered Nd-Fe-B and Sm-Co magnets [2]. Based on the grain boundary types present in the system, appropriate thermomechanical processing routines need to be applied to enable the wetting of grain boundaries by infiltrators. Hence, correlative microscopy techniques are employed to understand the role of grain boundary misorientation on the wettability and diffusion of multi-component low-melting eutectics that decorate the grain boundaries without reacting with the parent phases. Based on the fraction of infiltrators present along the grain boundaries and their local chemical compositions, the grain boundary energies of the investigated sites would be estimated, which is of fundamental importance towards developing grain boundary engineered high-performance permanent magnets.

References

1. Popov, A.G.; Golovnia, O.A.; Gaviko, V.S.; Vasilenko, D.Y.; Bratushev, D.Y.; Balaji, V.I.N.; Kovács, A.; Pradeep, K.G.; Gopalan, R.: J. Alloy Compd. 820 (2020) 1.
2. Glienke, M.; Vaidya, M.; Gururaj, K.; Daum, L.; Tas, B.; Rogal, L.; Pradeep K.G.; Divinski, S.V.; Wilde, G.: Acta Mater. 195 (2020) 304.

Max Planck partner group: Designing damage tolerant functional oxide nanostructures

Group head: B. N. Jaya

Indian Institute of Technology Bombay, Powai, India

Most functional microelectronic devices use ferroelectric ceramics like Barium Titanate ($BaTiO_3$) in nanostructured thin film form, while they suffer from brittleness at bulk-scale. In this study, we aim to enhance the damage tolerance of such material systems. Strategies can include exploiting size and/or interface effects. Prediction of damage tolerance and design of appropriate testing techniques for validation are carried out through numerical simulations.

In cooperation with C. Kirchlechner and G. Dehm from the SN department, micropillar compression tests of single crystal $BaTiO_3$ were performed. The experiments revealed a size effect in yield, with pillars <1 µm diameter reaching near theoretical strengths (Fig. 1), with size exponent close to -1 [1]. As a consequence, the elastic strain limit also significantly increased. This is extremely important for sensor/actuator applications in which the material constituting the device has to be defect-free and yet in its nanostructured form perform better than its bulk counterpart. In addition, a clear enhancement in damage tolerance was seen, with a transition from fracture to slip induced plasticity as the strain accommodating mechanism.

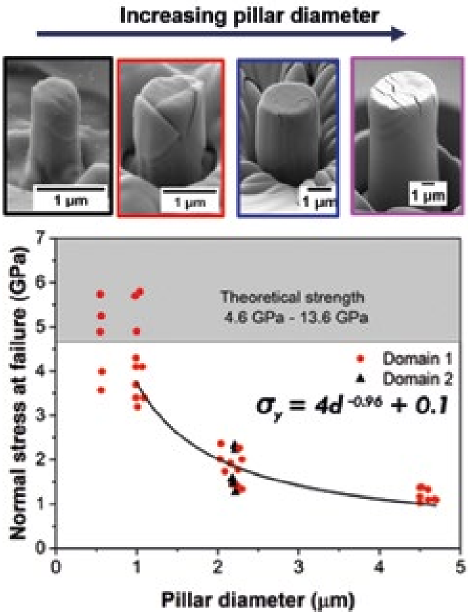

Fig. 1: *Strength and strain tolerance displayed by single crystal micropillars of $BaTiO_3$ as a function of specimen size.*

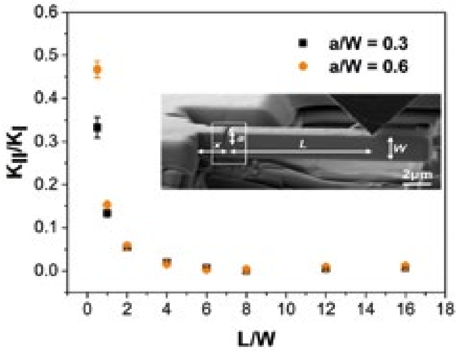

Fig. 2: *Microcantilever fracture test geometry and simulated mode mixity ratios as a function of beam aspect ratio and crack location.*

Failure in thin films attached to substrates will be through fracture or delamination. In films attached to the substrate, the highest mismatched system ($Si/BaTiO_3$) with highest tensile residual stress showed the highest crack density while the one with the best compatibility ($SrTiO_3/BaTiO_3$) showed the least crack density. The quantification of damage tolerance requires microscale fracture tests [2-3]. To overcome lack of standards and develop an artifact-free testing protocol for a microcantilever fracture test, simulations were carried out for varying beam dimensions and crack aspect ratios (Fig. 2) [4]. This was deployed to measure the fracture toughness (K_{IC}) of single crystals and thin films. K_{IC} of the free-standing thin film counterpart was found to be 75% lower than the single crystal, while films with residual stresses are currently being measured using bi-layered microcantilevers, which require crack driving force solutions that account for shielding and anti-shielding effects.

Understanding the impact of interface adhesion and residual stresses on damage tolerance using micro-scale testing techniques, followed by elastic strain engineering of these thin films is part of the ongoing work.

References

1. Mathews, N.G.; Saxena, A.K.; Kirchlechner, C.; Dehm, G.; Jaya, B.N.: Scr. Mater. 182 (2020) 68.
2. Chaudhari T.S.; Mathews, N.G.; Mishra, A.K.; Sahasrabuddhe, H.P.; Jaya, B.N.: JOM 73(6) (2021) 1597.
3. Mishra, A.K.; Lambai, A.; Jayaram, V.; Jaya, B.N.: Theor. Appl. Fract. Mech. 105 (2020) 102409.
4. Mathews, N.G.; Mishra, A.K.; Jaya, B.N.: Theor. Appl. Fract. Mech. (2021), accepted.

Max Planck partner group: Environmental degradation of high-temperature materials in service conditions

Group head: S. K. Makineni

Department of Materials Engineering, Indian Institute of Science Bangalore

The group's focus lies in exploring and understanding the atomic-scale degradation behaviour of γ/γ′ Ni-based superalloys and new CoNi based superalloys exposed to severe/harsh environmental conditions at high temperatures. Furthermore, the role of deformation-induced defects on the degradation will also be examined, in order to find material design routes to slow down or suppress aspects of the degradation and extend the lifetime of parts, i.e. with an eye on making engineering parts more sustainable.

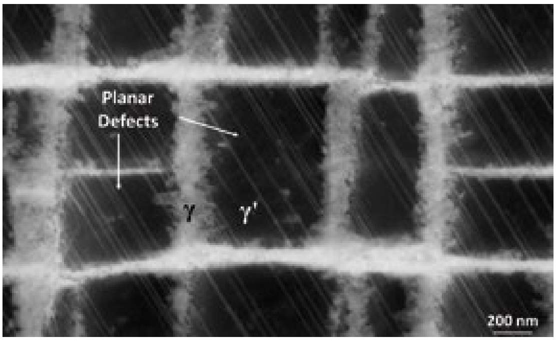

Fig. 1: *An ECCI image of CMSX-4 single crystal Ni-base superalloy after creep deformation at 800 °C under a stress of 800 MPa. It shows bright contrast regions corresponding to planar defects inside cuboidal γ′ precipitates.*

Gas turbine engines power passenger/military aircraft, ships, and power plants. High-temperature materials used in the engines experience extreme operational environments with highly pressurized gases at temperatures up to 1600 °C, i.e. higher than the melting temperature of the material used for the engine components. Combined usage of cooling systems and thermal barrier coatings, more specifically on blades, prevents them from melting [1]. However, the gaseous mixture consisting of hot water vapor (steam) containing sodium, sulphur, and halides intensifies and accelerates the corrosion of the components and causes rapid consumption and degradation of materials, eventually leading to the engine's catastrophic failure [2].

Commercial materials used for engine blades and disks are Ni-based superalloys in single- and poly-crystalline form [3]. These complex alloys contain over ten elements and exhibit a dual-phase microstructure comprising a face-centred-cubic (fcc) γ matrix phase and strengthening $L1_2$ ordered γ′ precipitates. During engine operation, the blades and disks go through stress-induced deformation, and accumulation of strain takes place in the material by the formation of a large number density of defects that includes dislocations, stacking faults (SFs) (as shown in Fig. 1), anti-phase boundaries (APBs) and micro-twins that are distinct in both chemistry (Fig. 2) and structure from the parent material [4–6].

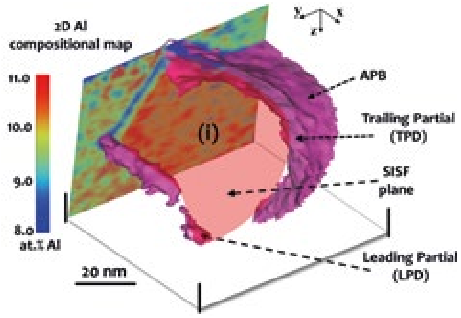

Fig. 2: *A 3D compositional map of planar defects comprising a stacking fault and an anti-phase boundary with their partial dislocations [5].*

Most studies were made on a macro-scale with a qualitative approach, leaving much room for precisely evaluating the effect of the role of individual defects (dislocations, SFs, APBs and micro-twins) on the material's corrosion behaviour, as it takes place dynamically during the engine's operation. The group's main aim is to explore and understand the atomic-scale degradation behaviour of high-temperature superalloys exposed to severe/harsh environmental conditions at high temperatures, the role of deformation-induced defects on the degradation, and define material design routes to slow down or suppress aspects of the degradation associated to oxidation, sulfidation, and corrosion at high temperatures. We use advanced microscopy and microanalysis, particularly the correlative use of electron microscopy and atom probe tomography to understand the mechanisms of atomic-scale environmental degradation of superalloys.

References

1. Padture, N.P.; Gell, M.; Jordan, E.H.: Science 296 (2002) 280
2. Eliaz, N.; Shemesh, G.; Latanision, R.M.: Eng. Failure Anal. 9 (2002) 31.
3. Pollock, T.M.: Nat. Mater. 15 (2016) 809.
4. Makineni, S.K.; Kumar, A.; Lenz, M.; Kontis, P.; Meiners, T.; Zenk, C.; Zaefferer, S.; Eggeler, G.; Neumeier, S.; Spiecker, E.; Raabe, D.; Gault, B.: Acta Mater. 155 (2018) 362.
5. Makineni, S.K.; Lenz, M.; Neumeier, S.; Spiecker, E.; Raabe, D.; Gault, B.: Scr. Mater. 157 (2018) 62.
6. He, J.; Zenk, C.H.; Zhou, X.; Neumeier, S.; Raabe, D.; Gault, B.; Makineni, S.K.: Acta Mater. 184 (2020) 86.

Max Planck partner group: Stress and defects-driven phase transformations

Group head: S. R. Meka

Indian Institute of Technology Roorkee, India

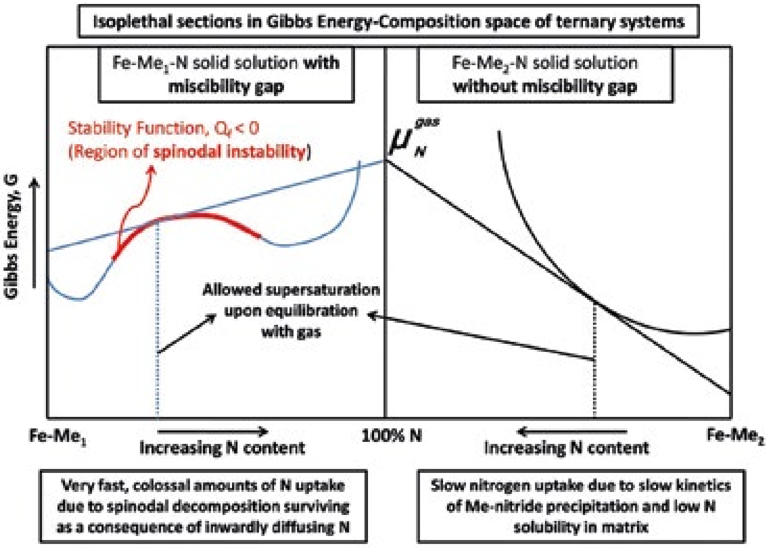

Fig. 1: *Schematic representation of the fundamental difference in thermodynamic properties of alloys exhibiting rapid and weak nitridation kinetics Me$_1$ represents alloying elements Al, Si and Mo and Me$_2$ represents alloying elements Cr, V and Ti. represents the chemical potential of N in the gas atmosphere.*

Phase transformations occurring in metallic solids exposed to chemical reservoirs of interstitial elements (H/C/N/O) are extremely important to understand in the context of thermo-chemical surface engineering, oxidation/dry corrosion of alloys and hydrogen storage materials. The main focus of the group has been to develop a fundamental understanding of such phenomena, strongly guided by CALPHAD-based, metastable gas-metal thermodynamic calculations. In the context of nitriding, several heretofore unrecognized effects of the presence of metastable miscibility gaps in alloy systems have been identified. Although the effect of different alloying elements on the nitridation kinetics of iron-based alloys has been extensively investigated experimentally, a theoretical explanation for the contrasting effects of different alloying elements, viz. rapid kinetics facilitated by Cr/V/Ti and slow kinetics allowed by Al/Si/Mo was previously unavailable. This has now successfully been explained by us by considering how nitrogen gets isothermally supersaturated into concentration regions of metastable immiscibility, followed by an isothermal phase-separation/spinodal-decomposition (Fig. 1) [1-2]. Consideration of the same phenomenon has also offered an explanation for the colossal levels of N/C supersaturation in stainless steels subjected to low temperature nitridation/carburization treatments [3-4]. Further, the cause for the rapidly passivating character of stainless steels at room temperature and the need for a minimum of about 11% Cr for such passivation was also related to the existence of a metastable miscibility gap in the Fe-Cr-O system [5].

In specific alloys undergoing nitridation, spinodal decomposition occurs while a N concentration gradient is present. Understanding the effect of misorientation between the direction of composition gradient and the crystallographically favourable direction for spinodal decomposition has led to a better explanation of the well-known grain-orientation-dependent N-uptake kinetics observed during nitriding of metallic alloys [6].

Another major research activity in strong collaboration with MPIE is focused at developing nitride-dispersion-strengthened steels by a novel powder processing route. Here, Al-alloyed steel powder is subjected to gaseous nitriding, which results in nanosized AlN in the ferrite matrix of steel powders [7], followed by the sintering or additive manufacturing of powders (Fig. 2) [8]. Recently, together with MPIE, we have started working on developing substitutional-interstitial HEAs by nitriding stainless steels [9] and medium entropy alloys.

References

1. Sasidhar, K.N.; Meka, S.R.: Acta Mater. 161 (2018) 266.
2. Sasidhar, K.N.; Meka, S.R.: Philos. Mag. 99 (2019) 2152.
3. Sasidhar, K.N.; Meka, S.R.: Scr. Mater. 162 (2019) 118.
4. Sasidhar, K.N.; Meka, S.R.: Sci. Rep. 9 (2019) 7996.
5. Sasidhar, K.N.; Meka, S.R.: Corros. Sc.i 166 (2020) 108433.
6. Sasidhar, K.N.; Gururajan, M.P.; Meka, S.R.: Philos. Mag. Lett. (2021) DOI: 10.1080/09500839.2021.1948130.
7. Yadav, A.S.; Kurnsteiner, P.; Jaegle, E.; Meka, S.R.: Powder Technol. 374 (2020) 527.
8. Yadav, A.S.; Kurnsteiner, P.; Jaegle, E.; Sasidhar, K.N.; Meka, S.R.: Steel Res. Int. (2021) DOI: 10.1002/srin.202100174
9. Qadri, S.A.R.; Sasidhar, K.N.; Meka, S.R.: Powder Technol. 390 (2021) 456.

Max Planck partner group: Microstructure design of high-performance materials for harsh energy applications

Group heads: J. Zavašnik[1,2], A. Šestan[1,2]

[1]Jožef Stefan Institute, Ljubljana, Slovenia; [2]Jožef Stefan International Postgraduate School, Ljubljana, Slovenia

The partner group on Microstructure Design of High-Performance Materials for Harsh Energy Applications has formally started the activities since June 1st 2021. The partner group evolved from the bilateral project "Investigation of helium retention in plasma facing materials using advanced analytical methods" between Jožef Stefan Institute and the department "Structure and Nano-/Micromechanics of Materials" at the Max-Planck-Institut für Eisenforschung, funded by Slovenian Research Agency and DAAD (2019-2020).

The research group will conduct innovative research on material – light elements interactions, a research field with relevance for nuclear and fusion reactors as well as for materials required for the hydrogen economy. The major aspects of research are studies on sinks for He and H in metals (e.g., precipitates, interfaces, grain boundaries), and their evolution at elevated temperature.

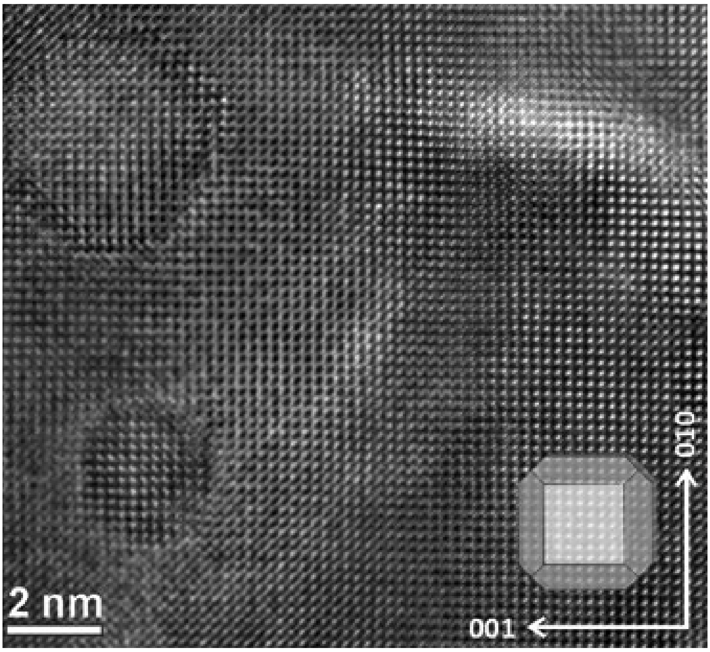

Fig. 1: *A transmission electron microscopy image of a W single-crystal with facetted He voids, interrupting the W crystal lattice. The voids are a combination of a cube {100} and rhombic dodecahedron {110}.*

The group's mission is to develop light-element damage tolerant microstructures of refractory materials (such as W and W-based alloys) by tailored structural imperfections, such as dislocations, voids, and grain boundaries. In the scope of the project, we aim to explore the transport, migration and coalescence of light elements at elevated temperatures while assessing the evaluation of the effects on the chemistry, microstructure and mechanical properties. Using state-of-the-art analytical tools and methods will unlock a unique insight into corrosion performance, hydrogen retention and permeability and behaviour of the materials at elevated temperatures. As a result, we aim to establish a fundamental understanding of the complex, synergistic, and inherently multiscale interactions in advanced energy materials down to the atomic level, when subjected to ion implantation and high temperatures, and to identify the interplay between microstructure and material properties.

References

1. Šestan, A; Zavašnik, J.; Maček, M.; Kocen, M.; Jenuš, P.; Novak, S.; Čeh, M.; Dehm, G.: J. Nucl. Mater. 524 (2019) 135.
2. Markelj, S.; Schwarz-Selinger, T.; Pečovnik, M.; Chrominski, W.; Šestan, A.; Zavašnik, J.: Nucl. Fusion 60 (2020) 106029.
3. Zavašnik, J.; Peng, J.; Palm, M.: Corros. Sci. 179 (2021) 109170.

Nano-/micromechanics of materials

Group head: J. P. Best

SN

Since April 2020, James Best leads both "Nano-/Micromechanics of Materials" and is interim group head for the "Thin Films & Nanostructured Materials" group. The scientific mission of the "Nano-/Micromechanics of Materials" group is to extract mechanical properties of materials at the microstructural length scale and link the mechanical response with the initial and evolving defect structure. The primary focus therefore lies on the deformation response of individual single crystals, bi-crystals or selected interfaces – where metallic thin-film systems are of particular interest.

To meet these aims, state-of-the-art methods such as focused ion beam milling and *in situ* scanning electron microscopy are required based on the requisite sample dimensions of <10 µm. The group oversees cutting-edge dual-beam electron microscopy infrastructure, combined with a suite of *in situ* characterization equipment allowing for highly resolved measurement of mechanical [1] and also electrical [2] properties.

Room temperature quasi-static mechanical tests are complimented by both high (800 °C) and low (-150 °C) temperature modules, along with high strain rate testing capabilities, which allow for detailed investigations of material behaviour under a range of environmental conditions. Insights into deformation, such as local fracture mechanics or dislocation plasticity, are then enriched through complimentary advanced characterization of structure and chemistry by colleagues at the MPIE. The role of local chemistry on the functional properties of materials is a growing theme in the group, with links to alloy design, corrosion and additive manufacturing [3, 4].

A broad spectrum of materials systems are under ongoing study; from advanced steels, tungsten alloys, to engineering oxides, topologically close packed intermetallic phases, or amorphous alloys. Materials are either produced at the MPIE using, for example, our physical vapor deposition cluster for metallic thin-films, or through our tight global network of processing groups at academic institutions and industry partners.

References

1. Ramachandramoorthy, R.; Yang, F.; Casari, D.; Stolpe, M.; Jain, M.; Schwiedrzik, J.; Michler, J.; Kruzic, J.J.; Best, J.P.: J Mater. Res. 36 (2021) 2325.
2. Luo, T.; Serrano-Sánchez, F.; Bishara, H.; Zhang, S.; Bueno Villoro, R.; Kuo, J.J.; Felser, C.; Scheu, C.; Snyder, G.J.; Best, J.P.; Dehm, G.; Yu, Y.; Raabe, D.; Fu, C.; Gault, B.: Acta Mater. 217 (2021) 117147.
3. Paul, M.J.; Liu, Q.; Best, J.P.; Li, X.; Kruzic, J.J.; Ramamurty, U.; Gludovatz, B.: Acta Mater. 211 (2021) 116869.
4. Best, J.P.; Ostergaard, H.E.; Li, B.; Stolpe, M.; Yang, F.; Nomoto, K.; Tarik Hasib, M.; Muránsky, O.; Busch, R.; Li, X.; Kruzic, J.J.: Addit. Manuf. 36 (2020) 101416.

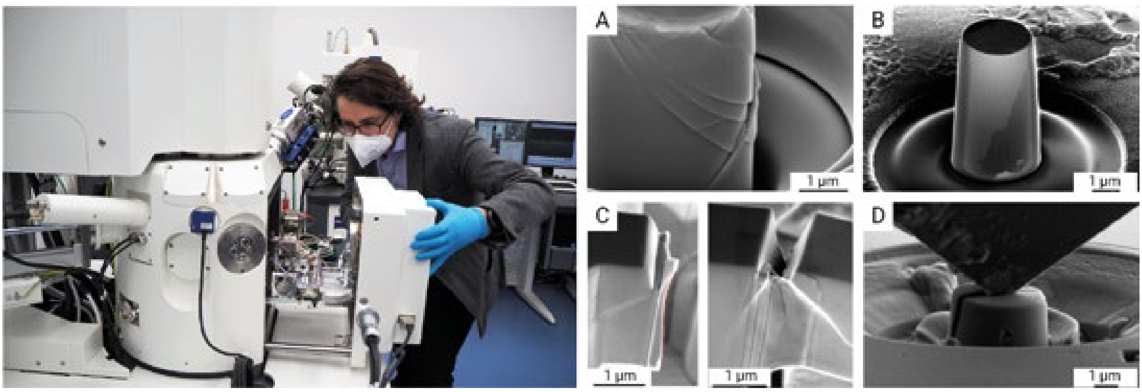

Fig. 1: *Combining broad materials science questions with advanced in situ nano- and micromechanical testing capabilities. Examples from the group's research include deformation in metallic glasses (A); copper bi-crystal milled from a thin-film containing an incoherent twin boundary (B); grain boundary fracture and plasticity in a tungsten alloy microcantilever (C); pillar-splitting fracture toughness testing of an intermetallic single crystal (D).*

Microstructure and mechanics

Group head: E. Bitzek

CM

The mechanical properties of materials are intrinsically determined by their microstructure – the entirety of crystal defects. The research group "Microstructure and Mechanics", established in September 2021, conducts research into the elementary defects of the crystalline lattice (e.g., dislocations, cracks, interfaces, point defects) and on how the organization and interaction of these defects influence material properties like strength, ductility, hardness, and fracture toughness as well as the material's behavior under fatigue, creep or tribological loading. To this end the group utilizes large-scale atomistic simulations with up to billions of atoms to obtain detailed information on defect properties and on defect – defect interaction mechanisms, see Fig. 1. The work of the group thus nicely complements the activities in the CM department that are focused on thermodynamic, kinetic and chemical aspects of defects. The group aims to improve our understanding and interpretation of experimental results, whilst simultaneously helping in the development of robust and reliable models for the prediction of deformation and failure of materials, like those developed in the theory and modeling group of the department MA.

One important approach to creating more realistic samples for atomistic simulations is the direct use of experimental information, e.g., from electron or atom probe tomography. Such "experimentally-informed" simulations have, e.g., revealed the synergistic effects between different strengthening mechanisms as well as the importance of precipitate curvature in dislocation – precipitate interaction in Ni-base superalloys.

A main focus of the group lies on how the properties of defects and defect-defect interactions are affected when the defects themselves contain defects, e.g., jogs on dislocations or crack fronts, or disclinations or disconnections in homo- or heterophase interfaces. Most computational studies of defects have so far been performed on idealized structures like infinite straight dislocations or crack fronts or perfectly planar interfaces that did not contain any additional defects. How the properties like their mobility or obstacle strength to dislocations of such idealized defects can be transferred to more realistic situations, including curved interfaces, is currently largely unexplored.

The simulation of nanomechanical tests offers a further way to directly link the simulations performed in the group to experiments. Nano-objects like thin films, nanowires and nanoparticles are usually defect free or contain only well-defined defects like twin boundaries and as such area ideal model systems for computational studies. By using large-scale atomistic simulations, the Microstructure and Mechanics Group studies size effects on elastic properties, dislocation nucleation-controlled deformation and small-scale plasticity at scales comparable to experiments as they are performed in the department SN.

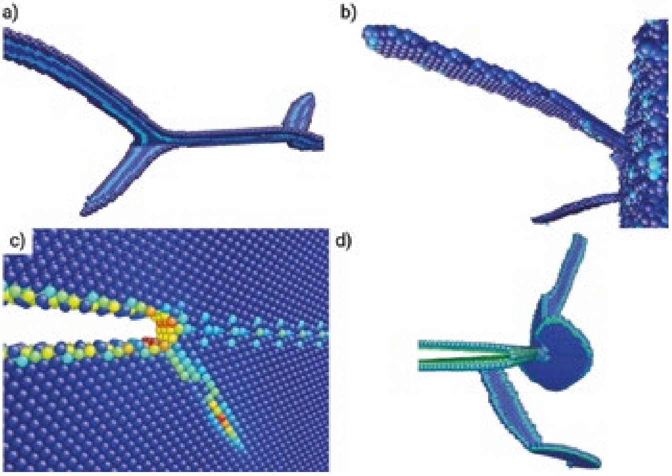

Fig. 1: *Examples of atomistic simulations of different defect-defect interactions: a) dislocation-dislocation interaction, b) dislocation-grain boundary interaction, c) grain boundary fracture and twin emission, d) dislocation – crack interaction and stimulated nucleation of dislocations.*

The group's activity also includes the modeling of mechanical properties of amorphous systems, including silica and meta-phosphate glasses, bulk metallic glasses, and polymers. Through his participation in the development of the Research Data Management (RDM) strategy of the Friedrich-Alexander-Universität Erlangen-Nürnberg (FAU) and being a founding member of NFDI (National Research Data Infrastructure Germany)-MatWerk, the group leader Erik Bitzek gained a deep expertise in RDM. By using this experience and maintaining an affiliation at FAU, he will support and strengthen the large-scale digitalization initiatives (NFDI, platform MaterialDigital) at the MPIE.

Atomistic modelling of material interfaces

Group head: T. Brink

SN

The research group Atomistic Modelling of Material Interfaces, headed by Tobias Brink, was established in 2020 with funding by the European Research Council (ERC, project GB-CORRELATE within the Horizon 2020 research and innovation programme). The main focus of the group is the investigation of grain boundary (GB) phases and their impact on material properties by means of atomistic computer simulations.

GBs affect many macroscopic properties of materials. In the case of metals, grain growth, Hall–Petch hardening, diffusion, and electrical conductivity [1], for example, are influenced or caused by GBs. It has been clear for some time that GBs can be treated as interface phases—existing only in contact with the abutting bulk phases—that can undergo phase transformations. These GB phases can have different properties and selecting specific GB phases could potentially provide an avenue to tailor macroscopic material properties. For this, information about the atomic structure of the GBs is required. This information is not only hard to resolve experimentally, but the great variety of possible GBs occurring in polycrystalline metals further complicates the process.

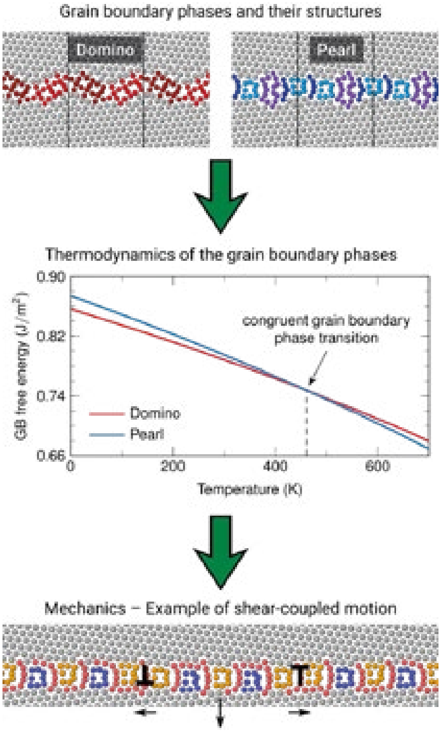

Fig. 1: *Example of the approach towards structure–property relations for GBs. First, the atomic structures of possible GB phases have to be modelled, followed by free energy calculations to understand phase stability and transitions. Finally, mechanical properties, such as the GB mobility, can be investigated for each GB phase.*

Our approach uses atomistic simulations to find the structures of specific GBs. The discovery of potential GB phases requires sampling of a complex phase space, which for reasons of computational efficiency is best done using classical molecular dynamics simulations with empirical potentials. An example of two GB phases obtained for the $\Sigma 37c$ ⟨111⟩ {1 10 11} tilt GB in copper [2] is shown in Fig. 1 (top). To ensure that the results of the computer models obtained with empirical potentials are reliable, we either employ density functional theory (DFT) calculations or we compare the structures to experimental data. Regarding the latter, we work closely with the experimentalists in the department to obtain high-resolution imaging data for tilt GBs [2].

Ultimately, the goal is to find engineering principles and structure–property relations for GBs [1]. Fig. 1 outlines the steps after obtaining and verifying GB phases. An important point is the derivation of phase diagrams for the GBs via free energy calculations to determine which phases can be synthesized experimentally. Regarding the mechanical properties, we are both interested in the movement of GBs, as well as their interaction with dislocations. Fig. 1 (bottom) shows an example of shear-coupled GB motion, where the motion is facilitated by disconnections (dislocation-like GB defects) specific to the GB phase.

The work of the research group is performed in close collaboration with the experimental activities at the institute, providing direct feedback loops: Insights from the simulation can be tested and applied on real materials and new experimental findings can be investigated in the computer in more detail.

This project has received funding from the European Research Council under the European Union's Horizon 2020 research and innovation programme (grant agreement No 787446).

References

1. Bishara, H.; Lee, S.; Brink, T.; Ghidelli, M.; Dehm, G.: ACS Nano 15 (2021) 16607.
2. Frommeyer, L.; Brink, T.; Freitas, R.; Frolov, T.; Dehm, G; Liebscher, C.H.: arxiv.org/abs/2109.15192 (2021).

Integrated computational materials engineering

Group head: M. Diehl

MA, now at KU Leuven (Belgium)

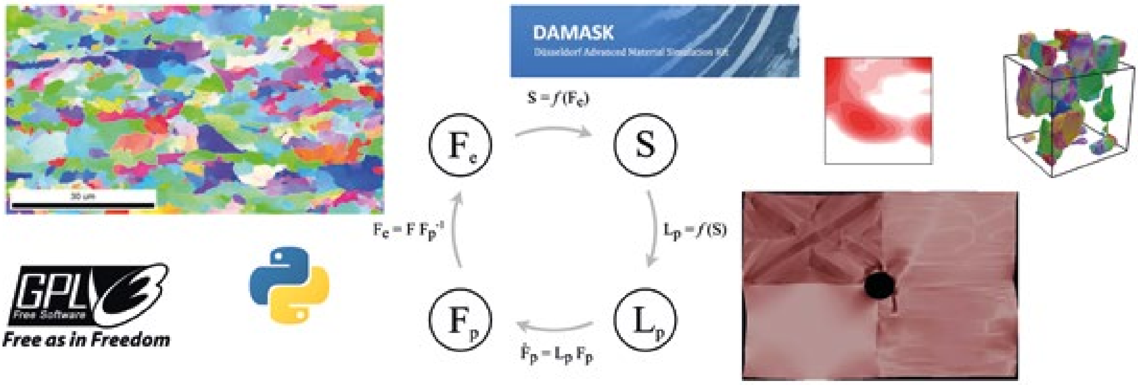

Fig. 1: *The research focus of the ICME group: Development of constitutive laws and numerical methods and integrating them into the free and open source package DAMASK to solve questions in materials science and engineering.*

The research group “Integrated Computational Materials Engineering” (ICME) was established in April 2019 and has its origin in the “Theory and Simulation” group headed by Dr. F. Roters. The aim of the ICME research group is the development, implementation, and application of models that allow to investigate how the materials respond to thermo-chemo-mechanical loads. To enable investigations at time and length scales relevant for engineering applications, the models are typically based on continuum approximations.

In order to have a software platform that allows to easily develop new models, the development and maintenance of the Düssseldorf Advanced Material Simulation Kit (DAMASK) is the key topic of the ICME group [1, 2]. DAMASK provides constitutive descriptions for (crystal) plasticity, damage, and temperature evolution that can be interfaced to external or internal solvers for boundary and initial value problems. As a free and open source software it is used in research groups from all over the world. The new DAMASK 3 integrates seamlessly into a larger ICME toolchain consisting for example of DREAM.3D, Neper, MTEX, Paraview and python-based analysis tools for data science.

To validate and apply the models implemented into DAMASK, we collaborate with experimentalists from the MPIE, other research institutions, and universities. This allows to employ the developed tools to answer materials science and engineering related questions. Moreover, experimental results are used to adjust the model parameters.

Besides the scientific work on enhancing DAMASK and applying it to solve questions in materials science and engineering, the enhancement of the user and developer documentation was an important task in the recent years. The new website (damask.mpie.de) provides now auto-generated documentation of all DAMASK tools and video tutorials for all relevant aspects of using DAMASK. M. Diehl is since October 2020 assistant professor for “Computational Materials Science” at the KU Leuven, Belgium. Intensive scientific exchange and co-supervision of PhD students [3, 4] are ongoing and are also planned for the longer future.

References

1. Roters, F.; Diehl, M.; Shanthraj, P.; Eisenlohr, P.; Reuber, C.; Wong, S.L.; Maiti, T.; Ebrahimi, A.; Hochrainer, T.; Fabritius, H.O.; Nikolov, S.; Friak, M.; Fujita, N.; Grilli, N.; Janssens, K.G.F.; Jia, N.; Kok, P.J.J.; Ma, D.; Meier, F.; Werner, E.; Stricker, M.; Weygand, D.; Raabe, D.: Com. Mater. Sci. (2019) 420.
2. Diehl, M.; Wang, D.; Mianroodi, J.R.; Han, F.; Ma, D.; Kok, P.J.J.; Roters, F.; Shanthraj, P.: Adv Eng Mater (2020) 1901044.
3. Gallardo-Basile, F.-J.; Naunheim, Y.; Roters, F.; Diehl, M.: Materials (2021) 691.
4. Sedighiani, K.; Diehl, M.; Traka, K.; Roters, F.; Sietsma, J.; Raabe, D.: Int. J. Plas. (2020) 102779.

Hydrogen mechanics and interfaces

Group head: M.J. Duarte Correa

SN

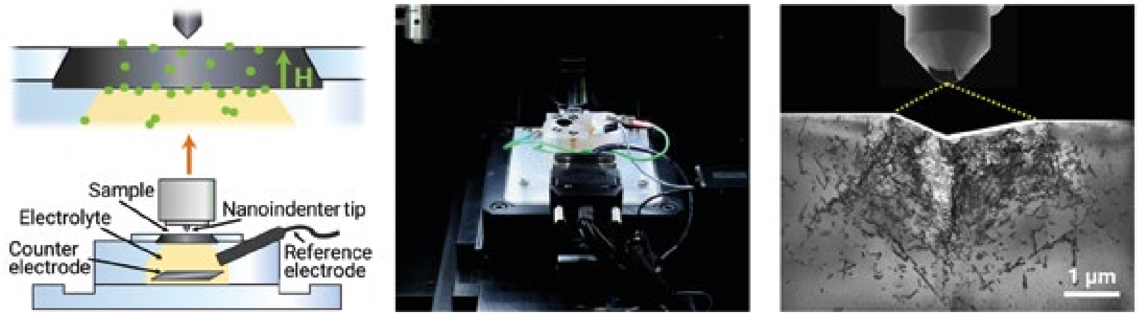

Fig. 1: *Novel back-side hydrogen charging setup developed in-house for in situ micromechanics during hydrogen charging and transmission electron microscopy image of a deformed microstructure after nanoindentation under the influence of hydrogen.*

This research group was established in September 2019 with the financial support of the European Research Council (ERC), aiming to understand the influence of hydrogen on the structure-property relations at the nanoscale, with particular interest at different interfaces.

While hydrogen is a promising candidate for the energy transformation and reduction of CO_2 emissions, it also represents a challenge as it might cause material degradation through hydrogen embrittlement. During hydrogen embrittlement several mechanisms leading to material failure can operate and lead to a sudden brittle fracture. The acting mechanisms are dependent on the hydrogen environment, the mechanical state of the structure and its particular chemical and microstructural features. Hence, to understand how hydrogen affects a particular material or which mechanism is dominant, it is necessary to study these interactions individually, thereby targeting analyses at the nano-/microscale.

Micromechanical testing through nanoindentation and related techniques (nanoscratching, pillar compression, diverse fracture toughness tests, etc.) are capable to reach the goal at the relevant size range. When mechanical testing is combined with direct hydrogen charging during deformation, the technique becomes a valuable asset to understand how failure initiates and then propagates due to hydrogen. M. J. Duarte Correa has developed a novel setup that combines these characteristics [1], and it is currently used and improved by the members of her group to unravel the role of hydrogen in materials deformation and failure (Fig. 1).

The research in the group is focused on different materials, ranging from model single-phase alloys to more complex steels, high temperature materials, high entropy alloys or hydrogen barrier coatings. Of special interest are interfaces, such as grain and phase boundaries, since they play a major role in hydrogen trapping and mobility, becoming preferential sites for material failure. By tailoring them through interface engineering and elemental segregation it is expected to improve the materials resistance to hydrogen.

The goals of the group can be summarized as follows:

- Develop unique dedicated instrumentation to understand hydrogen interactions in materials, with high-resolution and time-resolved measurements.
- Decipher failure mechanisms, quantify and model the hydrogen impact on specific mechanical behaviour.
- Provide essential data for modelling and simulations.
- Minimize hydrogen deleterious effects by enhancing properties and long-term-stability of materials in service (safer bridges, pipelines, reactors, planes, etc.).
- Provide guidelines for hydrogen tolerant materials, hydrogen storage and transport (safer and more efficient tanks) and fuel cell technologies.

To reach these goals the group has established multidisciplinary collaborations with all the departments at the MPIE and it is expanding its network to other universities, research centres and industry.

This project has received funding from the European Research Council under the European Union's Horizon 2020 research and innovation programme (grant agreement No 787446).

References

1. Duarte, M.J.; Fang, X.; Rao, J.; Krieger, W.; Brinckmann, S.; Dehm, G.: J. Mater. Sci. 56 (2021) 8732.

Thermodynamics and kinetics of defects

Group head: L. Huber

CM

Just as the rudder controls the direction of even a large vessel, defects (dislocations, grain boundaries (GBs), phase boundaries, etc.) strongly impact the bulk properties of a material. In the same way that the wheel of the ship in turn controls the rudder, alloying elements and chemical impurities play an out-sized role in determining defect behaviour – even when these chemical defects exist at extremely low nominal concentrations. This is because favourable interactions between structural and chemical defects can lead to large amounts of local enrichment. Understanding the interaction and co-evolution of these defects at the nano-scale is a central theme in the Thermodynamics and Kinetics of Defects (TKD) group.

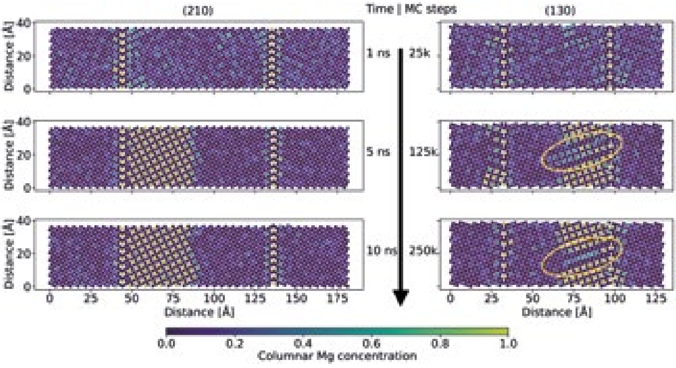

Fig. 1: *Coupled molecular dynamics/Monte Carlo simulations showing how grain boundary geometry in Al can promote the formation of a pristine Mg-rich phase, or impose persistent defects (circled yellow) in the secondary-phase. Al shown dark, Mg shown light, with columnar chemistry averaged along the GB tilt axis.*

Using atomistic simulations, the group strives to answer questions like: what does slip look like in complex phases [2]? What is the impact of GB structure on the nucleation and growth of precipitates [3] (Fig. 1)? How does solute segregation impact fracture at inter-phase boundaries? How does GB phase evolution differ under fast and slow mechanical loading conditions? In the same way that bulk phase diagrams were a key feature of materials engineering in the 20th century, a critical aspect of alloy design in the 21st century will be engineering at the nano-scale – guided by an understanding of defect phases and mechanisms [1]. Research in the TKD group works towards the overarching goal of understanding the various states defects can assume and how they evolve, to predict material behaviour, help interpret experimental results, and build per-defect phase diagrams.

Due to the large domain sizes needed to capture the structural complexity of realistic defects [4] and the combinatoric cost of exploring compositional space, i.e. the chemical decoration pattern at the defects, the main tool of the TKD group is classical molecular dynamics. Because of the complexity of the simulated systems, we regularly implement, improve, or develop from scratch state-of-the-art computational approaches for nano-scale modelling. The TKD group also develops concurrent multiscale approaches and machine-learned potentials, which both exploit highly accurate quantum-mechanical forces and energies from density functional theory. In addition to the challenges of chemo-structural complexity, accurately predicting thermodynamics and kinetics also requires a good treatment of thermal effects. To this end, the approaches we employ path sampling methods for capturing the non-local entropic effects of kinetic transitions, and thermodynamic integration approaches to capture the impact of thermal excitations on bulk and defect free energies.

These scientific activities are carried out within the framework of the CM's department's in-house software platform, pyiron [5]. Because of the high level of synergy between making advances in simulation methodology and building underlying computational infrastructure, pyiron development is a core part of the TKD group's mandate. In addition to atomic-scale materials science, the group also includes software engineering activities: extending scientific data management, the creation of a graphical user interface for pyiron, and the adding new for experimental materials science into pyiron, e.g. for data analysis.

References

1. Korte-Kerzel, S.; Hickel, T.; Huber, L.; Raabe, D.; Sandlöbes-Haut, S.; Todorova, M.; Neugebauer, J.: Int. Mater. Rev. (2021). DOI: 10.1080/09506608.2021.1930734

2. Guénolé, J.; Mouhib, F.Z.; Huber, L.; Grabowski, B.; Korte-Kerzel, S.: Scripta Mater. 166 (2019) 134.

3. Zhao, H.; Huber, L.; Lu, W.; Peter, N.; An, D.; de Geuser, F.; Dehm, G.; Ponge, D.; Neugebauer, J.; Gault, B.; Raabe, D.: Phys. Rev. Lett. 124 (2020) 106102.

4. Huber, L.; Hadian, R.; Grabowski, B.; Neugebauer, J.: npj Comput. Mater. 4 (2018) 1.

5. www.pyiron.org

Dynamic electrocatalytic interfaces

Group head: O. Kasian

GO, now also at: Helmholtz-Zentrum Berlin, Material Transformations in Electrocatalysis

Understanding the fundamentals of electrocatalytic reactions is of importance for the development of efficient and durable energy conversion and storage devices. In such devices electrocatalyst materials are used to accelerate the target reactions, in which only topmost atoms contribute to the electrocatalysis. In time these surface atoms undergo transformations, e.g. oxidation, poisoning or even dissolution, which affects further performance of energy conversion and storage devices. Understanding the evolution of the topmost atomic layers plays crucial role for the development of durable and stable electrocatalysts. This is, however, challenging task which requires implementation of advanced electrochemistry methods in combination with surface characterization techniques, ideally with atomic level resolution [1].

The guest group 'Dynamic Electrocatalytic Interfaces' develops nanostructured model thin film surfaces and explores their structural and compositional changes induced by the catalytic processes and the effect of such transformations on the mechanism and kinetics of the reactions in electrocatalysis. This is achieved by combination of various ex situ and in situ methods. The main research task is to provide a detailed mechanistic understanding of chemical processes ongoing at complex and dynamic interfaces, with a special focus on electrocatalysis for energy conversion and storage, aiming to overcome stability limits of the catalyst materials [1-3]. The scope of methods is schematically shown in Fig. 1 and includes variety of physical vapor deposition techniques (PVD), advanced electrochemical characterization methods, atom probe tomography (APT) and X-ray photoelectron spectroscopy including synchrotron based methods. The group is funded by the Helmholtz Zentrum Berlin and Helmholtz Association and works in close cooperation with the MA department and 'Nanoanalytics and Interfaces' group.

References

1. Kasian, O.; Geiger, S.; Li, T.; Grote, J.P.; Schweinar, K.; Zhang, S.; Scheu, C.; Raabe, D.; Cherevko, S.; Gault, B.; Mayrhofer K.J.J.: Energy Environ. Sci. 12 (2019) 3548.
2. Kasian, O.; Li, T.; Mingers, A.M.; Schweinar, K.; Savan, A.; Ludwig, A.; Mayrhofer, K.J.J.: J. Phys. Energy 3 (2021) 034006.
3. Schweinar, K.; Gault, B.; Mouton, I.; Kasian, O.: J. Phys. Chem. Lett. 11(13) (2020) 5008

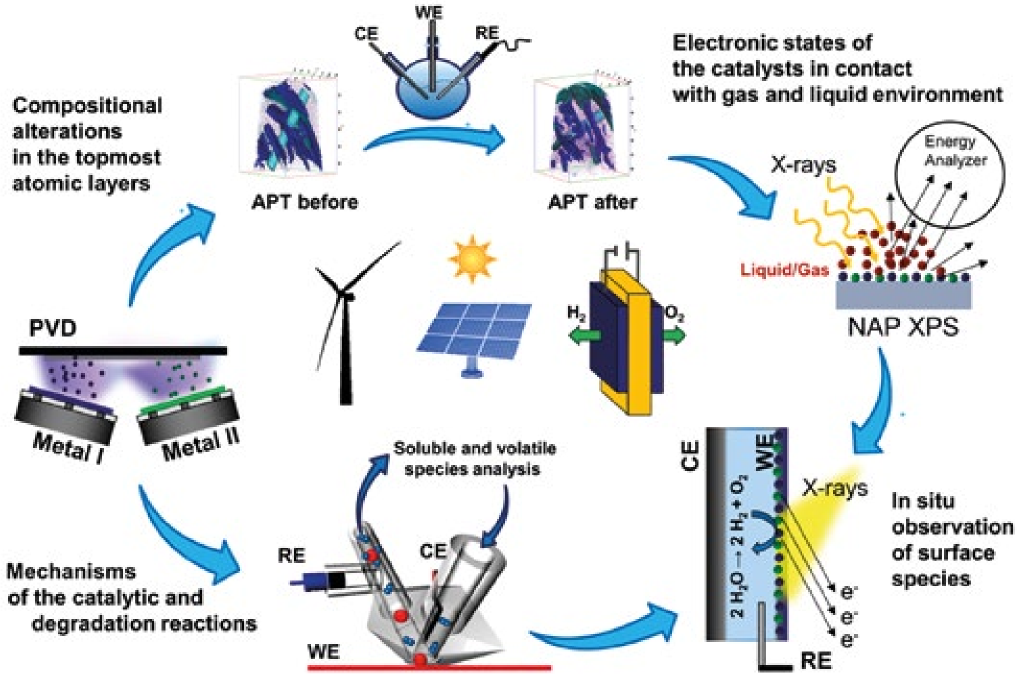

Fig. 1: *Schematic presentation of the approach to achieve the goals of the group 'Dynamic Electrocatalytic Interfaces.'*

High performance alloys for extreme environments

Group head: P. Kontis

MA, now also at Norwegian University of Science and Technology

There is an increasing need of more efficient and environmentally friendly aero-engines and land-based gas turbines, that can be addressed by higher operational temperatures or alternative fuels, such as hydrogen. Thus, new advanced alloys with enhanced performance in extreme environments are vital to our society and its sustainable future.

Therefore, the research group "High performance alloys for extreme environments" was established in July 2020. Our group aims to decipher the fundamental physics governing the partitioning and diffusion of solutes at crystal defects during plastic deformation under creep and fatigue conditions in aerospace materials, such as Ni-based superalloys or Ti-alloys, so as to guide the design of new alloys with enhanced properties.

We exploit a framework of high-end microscopy and microanalysis, in particular atom probe tomography and electron microscopy (scanning and transmission) to provide fundamental insights into how, why and what solutes interact with crystal defects (dislocations, stacking faults) in high performance alloys. These interactions of solutes with defects result in the so-called "plasticity-assisted redistribution of interacting solutes mechanisms" at the near atomic scale. As a consequence, local chemical inhomogeneities and undesirable microstructural alterations are promoted that lead to fracture. The partitioning of a solute at a crystal defect depends on the type of crystal defect, the deformation conditions, i.e. temperature and stress and the overall alloy composition. By understanding these interactions of solutes with defects, we aim to provide a fundamental understanding of the structure-chemistry relationship at near-atomic level. We then use these new insights to develop alloy design strategies for advanced alloys for extreme environments.

For instance, we have recently revealed the partitioning of solutes at stacking faults in a Ni-based superalloy after creep at 750 °C, similar to those shown in Fig. 1. By manipulating the alloy composition, thus the solutes partitioning at the stacking faults, we promoted a local phase transformation mechanism along the faults, that hinders the shearing of the main strengthening precipitates and lowers the deformation creep rate of the alloy. These findings allowed us to suggest an alloy design guideline through which this local phase transformation mechanism is promoted [1]. Lowering the deformation rate results in extending the life of components and thus making gas turbines more sustainable.

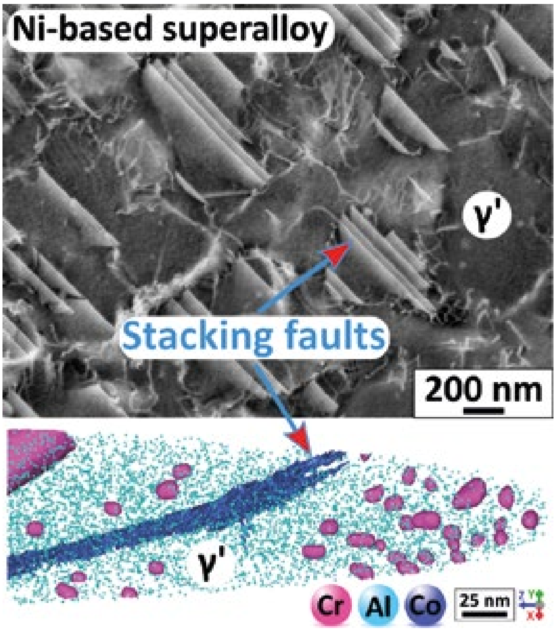

Fig. 1: *Electron channelling contrast image of a deformed superalloy after creep at 750 °C showing stacking faults in γ' precipitates, alongside a 3D-atom probe reconstruction revealing segregation of cobalt at stacking faults.*

Our group also aims to understand the role of hydrogen on the performance of advanced alloys, aiming to enable the use of hydrogen as a fuel in engines. Although, hydrogen embrittlement is known to reduce significantly the ductility of superalloys at room temperature, it is not clear what the exact effect of hydrogen is at service temperatures (above 600 °C) and also during cyclic thermo-mechanical loading. Hydrogen is often assumed to segregate in dislocations, but segregation of other solutes, such as chromium and cobalt [2], at service temperatures may influence the detrimental role of hydrogen in the mechanical performance of advanced alloys. Thus, the interactions of solutes with crystal defects and their role in the performance of advanced alloys becomes a very critical future alloy design parameter [3].

References

1. Lilensten, L.; Antonov, S.; Gault, B.; Tin, S.; Kontis, P.: Acta Mat. 202 (2021) 232.
2. Kontis, P.; Zhuangming, L.; Collins, D.M.; Cormier, J.; Raabe, D.; Gault, B.: Scripta Mat. 145 (2018) 76.
3. Kontis, P.: Scripta Mat. 194 (2021) 113626.

Computational sustainable metallurgy

Group head: J. R. Mianroodi

MA

The “Computational Sustainable Metallurgy” group, established in 2020, focuses on the application and development of computational methods to solve various challenges related to materials and processes with significant environmental impacts. These are problems in which the interplay of chemistry, phase transformation, microstructure, mechanics and damage plays an important role. Iron ore reduction with hydrogen, hydrogen storage, and battery technology are some of the focus areas in this group. The development of novel methods for multi-scale multi-physics modelling of complex material systems with machine learning, as required for the challenging materials science issues related to sustainability, is part of the activities in this group.

The use of green hydrogen as an alternative reducing agent to carbon for iron production will lead to a significant reduction in CO_2 emissions in the industrial sector. Despite the development and availability of a range of other construction materials, steel remains an important factor in the global economy. For example, annual global steel production of 1700 million metric tons (Mt) in 2019 far exceeds that of aluminium (94 Mt), the second most widely produced metal. Global steel production of this magnitude accounts for about 6 % of global energy consumption and nearly 25 % of industrial CO_2-equivalent emissions [1]. As the EU is the world’s second largest steel producer, a major contribution to achieving the Green Deal must be a significant increase in energy efficiency and a substantial reduction in CO_2 (and other) emissions from steel production.

The reduction process of iron ore involves chemical reactions, phase transformation, mechanical deformation and damage (porosity) development on different length and time scales [2]. For example, the reduction of iron ore to iron results in a volume change of 40 %, which leads to the build-up of residual stresses, inelastic deformation, and pore formation. The latter, in turn, strongly affects the overall reaction/reduction rate and process kinetics. Given these multi-physics conditions, the modelling work in this group is based on finite deformation phase-field chemo-mechanics (PFCM) [3]. For application to iron ore reduction modelling, the methodology is extended to include chemical reactions and surface catalysis effects between gas and solid. These calibrated models are applied to determine optimal reduction conditions for each case.

Machine learning (ML) is also used in this group to accelerate the complex numerical solvers as well as to bridge the scale gap from microstructure to macroscopic material behaviour. The ML-based approach has been shown to be up to three orders of magnitude faster than the conventional spectral mechanical solver [4]. The increased performance enables more advanced multi-scale multi-physics modelling, which is necessary for many sustainability related material science challenges.

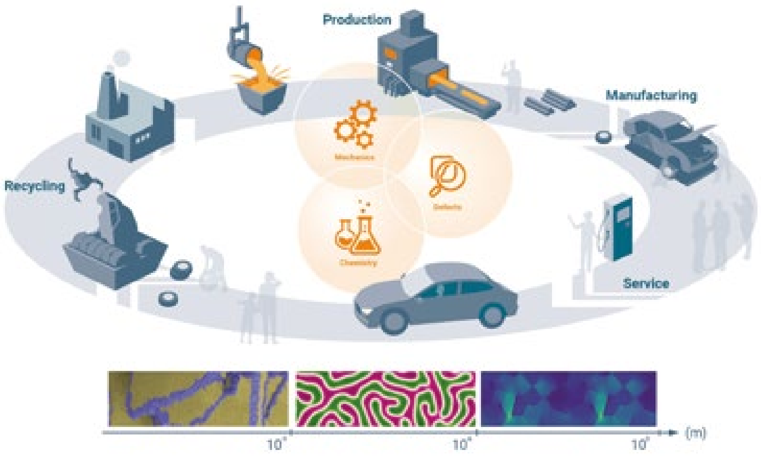

Fig. 1: *Schematic of metals’ life-cycle. Investigating this cycle requires understanding of chemistry, mechanics and microstructure. The theoretical and computational study of new low-carbon footprint alternatives in metal production and recycling is the focus of this group.*

References

1. Raabe, D.; Tasan C. C.; Olivetti E. A.: Nature 575 (2019) 64-74.
2. Kim S.; Zhang X.; Ma Y.; Souza Filho I.R.; Schweinar K; Angenendt K; Vogel D.; Stephenson L. T.; El-Zoka A. A.; Mianroodi J. R.; Rohwerder M.; Gault B.; Raabe D.: Acta Mater. 212 (2021) 116933.
3. Svendsen B.; Shanthraj P.; Raabe D.: J Mech. Phys. Solids 112 (2018) 619.
4. Mianroodi, J. R.; Siboni, N. H.; Raabe, D.: npj Comput Mater 7 (2021), 99.

Spectroscopy at electrochemical interfaces

Group head: M. Rabe

GO

The early career research group ‘Spectroscopy at Electrochemical Interfaces’ was established in 2020 as joint research group of the MPIE and the cluster of excellence RESOLV (www.solvation.de). The group studies fundamental properties and dynamic processes on solid interfaces using optical interface spectroscopy methods.

The properties of solid surfaces can be conveniently adjusted and functionalized with polymers, biomolecules and self-assembled monolayers (SAMs). One focus of our group is to control and study such interfaces and their solvation, that is the structure of the solvent layers that form at the solid/liquid junction. Since any interface reaction is accompanied by structural changes of the solvation layers, a thorough understanding of these phenomena is needed for the design of new improved materials with increasing inertness to corrosion, bioadhesion and -fouling, or to design new ‘smart’, i.e. switchable interfaces. For instance, the potential induced switching of the germanium interface termination, triggers the desolvation of the interface [1]. This switching is employed as a hydrophobic/hydrophilic switchable ‘smart’ interface to trigger reversible adsorption as well as reversible reorientation of biomacromolecules. Such switchable interfaces may find applications as highly functional biosensors. Furthermore, research is done to understand the influence of nanoscale structures and chemistries on the solvent structure. To that end, SAMs are a convenient tool for designing interface properties on the nanoscale. Nanoscale phase separated hydrophobic/hydrophilic SAMs will be studied by resonance enhanced atomic force microscopy IR spectroscopy (RE AFM-IR), a method that allows interface spectroscopy with resolution in the region of ~10 nm. Experiments in humidified atmosphere will allow to study the structure of adsorbed ultrathin water layers that form on these interfaces. The aim is to obtain local structural information of water adsorbed on nanostructured interfaces. Another approach is the study of gold nanoparticles by means of solvation shell attenuated total reflection-infrared (ATR-IR) spectroscopy. This method includes multivariate data analysis approaches for which tools are developed in the group [2].

A second main focus of the groups research is the study of electrocatalytic water splitting for production of green hydrogen. The anodic partial reaction, the oxygen evolution reaction (OER), is sluggish and materials based on earth-abundant first row transition metals such as Co, Mn, Fe, Ni are widely studied as low-price catalytic materials, for the industrial scale application. An in-depth understanding of the catalytic mechanisms as well as on degradation mechanisms that occur on these electrodes can be obtained by *operando* and *in situ* spectroscopies and is necessary to guide the knowledge-based material design. Operando Raman spectroscopy and spectroscopic ellipsometry on Mn-oxides that formed on metallic substrates, have revealed phase transitions and strong dissolution occurring during OER, which is a generic problem for their widespread application [3]. In a novel project that is part of a cluster project funded by the BMBF (Federal Ministry of Education and Research) ‘Ideenwettbewerb Wasserstoff’, these methods are applied and further extended to study porous Ni based electrodes under elevated current and temperature conditions that are more relevant for industrial alkaline water electrolysis. In parallel, operando surface enhanced Raman spectroscopy (SERS) and infrared spectroscopy (SEIRAS) will be established on Ni substrates for studying thin catalyst films which can yield structural information about the catalytic active centers and intermediates.

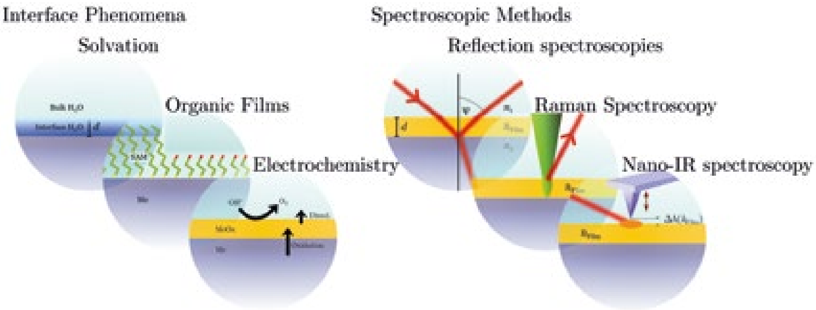

Fig. 1: *Studied interface phenomena and methods employed in the group.*

References

1. Niu, F.; Rabe, M.; Nayak, S.; Erbe, A.: J. Chem. Phys. 148 (2018) 222824.
2. Rabe, M.: J. Open Res. Softw. 8 (2020) 13.
3. Rabe, M.; Toparli, C.; Chen, Y.-H.; Kasian, O.; Mayrhofer, K. J. J.; Erbe A.: Phys. Chem. Chem. Phys. 21 (2019), 10457.

Nanomechanical instrumentation and extreme nanomechanics

Group head: R. Ramachandramoorthy

SN

The "Nanomechanical Instrumentation and Extreme Nanomechanics" (XNano) group of Rajaprakash Ramachandramoorthy was established in 2020. It aims at addressing the two fundamental bottlenecks of micro/nano-mechanics research: the lack of appropriate small-scale testing platforms capable of mechanical metrology under application relevant extreme strain rates/temperatures [1] and small-scale sample fabrication technologies capable of manufacturing ideal test-beds for statistically relevant mechanical testing [2].

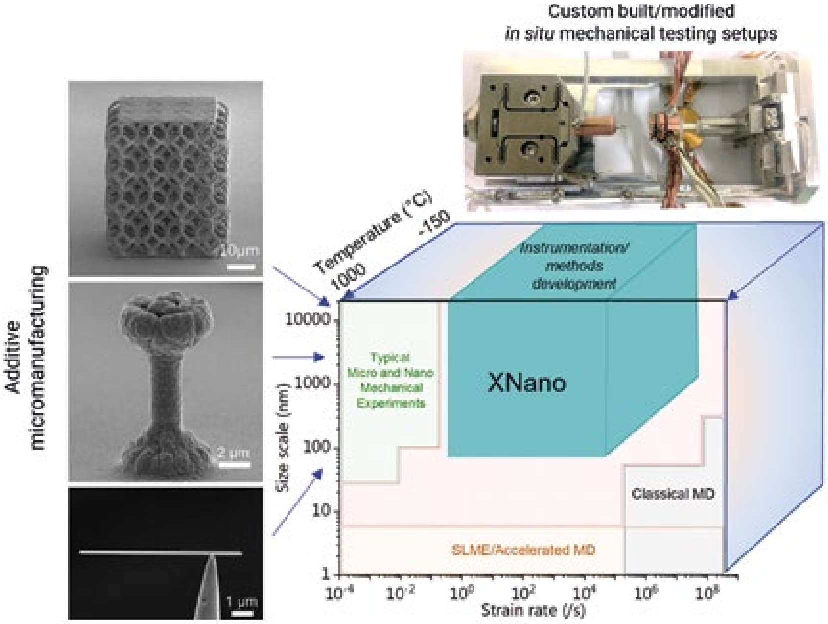

Fig. 1: *Summary figure showing the core foci of the XNano group in pushing the boundaries of small-scale mechanics towards higher strain rates (and high/cryo temperatures) and small-scale manufacturing towards complex 3D architectures.*

The relentless pursuit towards miniaturization of materials has resulted in micro/nano-scale materials becoming ubiquitous functional materials in a wide variety of application including microelectronics and watchmaking. Given that these small-scale materials can be subjected to day-to-day mechanical and thermal overloads such as drops, impacts and vibrations, the understanding of their dynamic thermomechanical properties is critical to assess their reliability and suitability in such applications. From an academic perspective, experimental determination of high strain rate properties at the micro- and nano-scale will allow the bridging of time scales between simulations (molecular dynamics/discrete dislocation dynamics) and experiments, enabling a direct one-on-one comparison between the two complementary research methodologies [3].

As such, the primary research focus is on instrumentation development for *in situ* nano-to-meso scale multimodal (tension, compression and fatigue) mechanical investigations at strain rates between 0.001/s to 10000/s and temperatures between -160 °C and 1000 °C. Specific case studies using such customized instrumentation are planned on small-scale architectures including nanowires, micropillars [4], microparticles and microlattices, which will help extract their structure (architecture) - microstructure - mechanical property relationships under extreme loading conditions. The second key area of research will be on the development of additive micromanufacturing methods for fabricating nano-to-meso scale metallic architectures. A localized electrodeposition based microadditive technology will be developed to print micro/nano-scale architectures, using different metals including copper, nickel and iron.

References

1. Ramachandramoorthy, R.; Schwiedrzik, J.; Petho, L.; Guerra-Nunez, C.; Frey, D.; Breguet, J.M.; Michler, J.: Nano Lett. 19(4) (2019) 2350.
2. Schürch, P.; Ramachandramoorthy, R.; Pethö, L.; Michler, J.; Philippe, L.: Appl. Mater. Today 18 (2020) 100472.
3. Ramachandramoorthy, R.; Gao, W.; Bernal, R.; Espinosa, H.: Nano Lett. 16 (1) (2016) 255.
4. Ramachandramoorthy, R.; Yang, F.; Casari, D.; Stolpe, M.; Jain, M.; Schwiedrzik, J.; Michler, J.; Kruzic, J.J.; Best, J.P.: J. Mater. Res. 36 (2021) 2325.

Gottfried Wilhelm Leibniz Prize 2020

B. Gault

MA

In 2020, I was awarded the Leibniz Prize of the German Research Foundation (DFG) for my frontier work in atom probe tomography (APT) and the research that the technique enables. After being postponed a few times, the ceremony took place remotely in November 2020 (Fig.1).

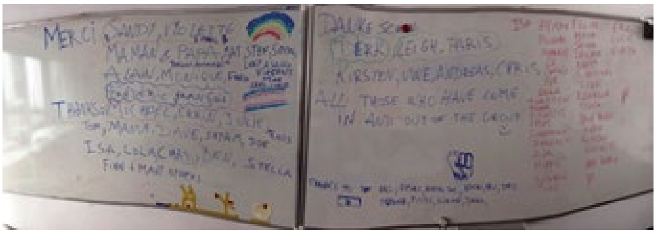

Fig. 1: *White board in my office that I used as background during the online ceremony in November 2020, thanking (almost) all those who helped and contributed to my adventures.*

I have been fortunate, over the course of my research career to work on almost all aspects of APT. I initiated during my PhD the implementation of an ultrafast laser source on an atom probe and deciphering the physics of the field evaporation process – which I did wrongly funnily enough. Yet, this work was a great enabler in the development of the technique, and pulse-laser sources are now implemented on nearly all commercial instruments. In the following years, as a postdoctoral scientist, I worked also on improving the approaches to build the tomographic reconstruction, and working on quantifying more precisely the spatial performance of the technique, and start using it to characterize engineering materials.

Since joining the MPIE in 2016, I cannot think of a single aspect of this previous experience that has not been useful. I have had a chance to renew my interests in instrument development with our cryo-enabled suite of instruments [1] and the Reacthub Module (see p. 58), data mining, in part via our involvement in the BIGMAX network (see p. 82) [2], the theory of field evaporation and field ionisation with the development of the analytical-field-ion microscope [3], and the very many materials science projects, with an emphasis on understanding the fundamental processes that take place in complex, engineering materials, incl. Al-alloys, steel, zirconium alloys, semiconductors amongst others. A focus has been on the use of APT to understand the relationships between their microstructure and properties, and their evolution over the course of their use in service, insights that are necessary to guide design of future materials with enhanced lifetime and improved sustainability.

Although the Prize is supposedly awarded to me, I would never have received it without the good fortune of working with amazing colleagues, doctoral students, postdoctoral and senior scientists and technicians, at the MPIE and in all institutions where I worked, from the University of Rouen, the University of Sydney, Oxford University, and McMaster. For me, MPIE epitomizes the collaborative scientific endeavour, and I'm extremely grateful to all colleagues here with whom we have managed to build fantastic joint studies involving other microscopy techniques, atomistic simulations and many materials systems – I have learnt so much.

So, what is next? I wish to keep pushing the use of atom probe to its limits, to better understand complex energy materials incl. materials for clean power generation (see p. 25). But the next frontier is to deploy the strength of atom probe to the analysis of liquids, liquid-solid interfaces and soft matter. Since the 1950s, several scattered investigations of biological materials have been reported, but without a sustained effort that would have enabled true breakthroughs. In parallel, the development of cryo-transmission electron microscopy has initiated a revolution in the biological sciences, enabling to image proteins and viruses at an unprecedented resolution. Atom probe could, here again, provide unique insights – an example is the presence of metallic species suspected to be involved in the Alzheimer disease. A few years ago, we started looking at such proteins, and the grant from the Leibniz Prize will allow me, in part, to push this research, via cryo-specimen preparation, transport and analysis of frozen liquids. The first forays are indeed already promising [4].

References

1. Stephenson, L.T.; Szczepaniak, A.; Mouton, I.; Rusitzka, K.A.K.; Breen, A.J.; Tezins, U.; Sturm, A.; Vogel, D.; Chang, Y.; Kontis, P.; Rosenthal, A.; Shepard, J.D.; Maier, U.; Kelly, T.F.; Raabe, D.; Gault, B.: PLoS One 13 (2018) 1.
2. Wei, Y.; Varanasi, R. S.; Schwarz, T.; Gomell, L.; Zhao, H.; Larson, D. J.; Sun, B.; Liu, G.; Chen, H.; Raabe, D.; Gault, B.: Patterns 2 (2021) 100192.
3. Katnagallu, S. S.; Stephenson, L. T.; Mouton, I.; Freysoldt, C.; Subramanyam, A. P. A.; Jenke, J.; Ladines, A. N. C.; Neumeier, S.; Hammerschmidt, T.; Drautz, R.; Neugebauer, J.; Vurpillot, F.; Raabe, D.; Gault, B: New J. Phys. 21 (2019) 12 123020.
4. El-Zoka, A. A.; Kim, S.-H.; Deville, S.; Newman, R. C.; Stephenson, L. T.; Gault, B.: Sci. Adv. 6 (2020) 49.

video recorded by the DFG and played during the ceremony.

ERC Consolidator Grant: Microscopic origins of fracture toughness

E. Bitzek

CM

The resistance to crack propagation is undoubtedly one of the most important properties of structural materials. However, our current mechanistic understanding of the fracture processes in typical semi-brittle materials like steels, refractory metals, or semiconductors is not sufficiently advanced to predict the fracture toughness K_{Ic} and its dependence on the microstructure, temperature, and strain rate. Therefore, K_{Ic} is commonly regarded as a phenomenological material parameter for fracture mechanics models that require experimental calibration. A quantitative understanding of the relation between physical, crystallographic and in particular microstructural properties (Fig. 1) and the failure resistance of a material is, however, crucial for improving the performance of materials and material models. Understanding crack-microstructure interactions is, furthermore, of fundamental importance for fatigue fracture, stress corrosion cracking and hydrogen embrittlement – topics which are of interest for all MPIE departments.

The aim of microK_{Ic} is to study fracture in model materials in order to gain a detailed understanding of the microscopic crack-tip processes during fracture initiation, propagation and arrest, and to systematically study the interactions of cracks with constituents of the microstructure like dislocations, voids, precipitates and grain boundaries. To this end, we perform fully 3D, large-scale atomistic simulations on cracks in bcc-based materials (Fig. 2) with varying crack orientation, crack front quality, and in the presence of dislocations and microstructural obstacles.

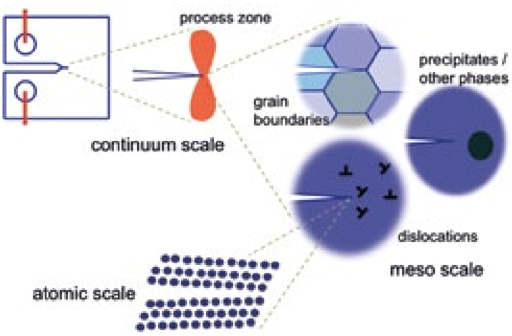

Fig. 1: *Relevant scales and microstructural features that determine the response of a crack to an external load.*

The criteria for crack advance and dislocation nucleation at crack tips obtained from atomistic simulations are being implemented in a newly developed coupled finite element - discrete dislocation dynamics (DDD) code. This DDD+Crack code has been developed together with Prof. M. Fivel (Université Grenoble Alpes, France) within microK_{Ic} and now allows for the first time fully 3D studies of fracture and crack-tip plasticity at the mesoscale.

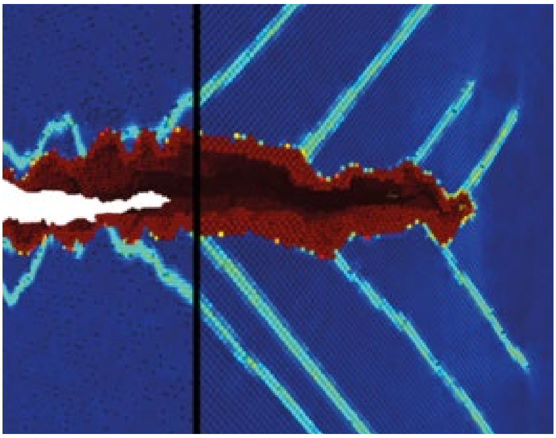

Fig. 2: *Part of a large-scale 3D fracture simulation in off-stoichiometric B_2 NiAl showing how atomic-scale heterogeneities can influence the crack propagation and dislocation emission (atoms coloured by atomic strain).*

In addition to the simulations, micromechanical fracture tests on notched cantilever beams with well-defined microstructures are being carried out at the Friedrich-Alexander-Universität Erlangen-Nürnberg, to which Erik Bitzek maintains close contacts. Close interactions with the micro- and nanomechanical testing activities in the SN department are also envisaged. The micromechanical experiments will be compared to the results of multiple DDD+Crack simulations with different microstructures. That way, the outcome of the simulations can be qualitatively validated.

The ultimate goal of microK_{Ic} is to use this experimentally validated multiscale modelling framework to develop a microstructure-sensitive, physics-based micromechanical model of the fracture toughness. In collaboration with the theory and modelling group of the MA department, such a model will be implemented in their CP-FEM code. In the long term, a better understanding of the microscopic origins of fracture toughness will contribute to the development of new, failure-resistant materials and help to improve design guidelines for safety-relevant structures and components.

This project has received funding from the European Research Council (ERC) under the European Union's Horizon 2020 research and innovation programme (grant agreement No 725483).

ERC Advanced Grant: Correlating the state and properties of grain boundaries

G. Dehm

SN

The project GB-CORRELATE [1], which started in August 2018, explores the presence and consequences of grain boundary phase transitions in pure and alloyed Cu and Al. If grain size gets smaller and smaller - like in nanocrystalline materials - the grain boundary (GB) volume can exceed several 10 % of the total material volume and become most important for the resulting properties. The atomic coordination and chemistry of such GBs may undergo phase transitions, abrupt changes in structure and chemistry, which will impact the material behaviour - like strength, thermal stability, electrical resistance - even for conventional materials. However, this interplay between GB phases and material properties is not yet understood. Experimentally, GBs are difficult to study - it needs atomic resolution and sensitivity with respect to chemistry. In addition, it is unknown under which conditions phase transformations of GBs occur. A fundamental understanding requires atomistic modelling connected with smart experiments.

One of our approaches is to grow Cu and Al films on (0001) oriented α-Al_2O_3 substrates, which led to a strong (111) texture with several symmetric and asymmetric tilt GBs [2]. Using Cu thin films we could show for the first time that a pure elemental metal which forms only a single bulk fcc phase as a solid can form several GB phases [3]. We observed at a ∑19b [111] tilt GB 3 different atomic motifs (phases) for different grain boundary inclinations. At asymmetric ∑19b [111] and symmetric ∑19b [111] $(17\bar{8})$ GB we found the coexistence of two GB phases [3] (Fig.1).

The coexistence of the two phases (Fig. 1) indicates a sluggish transition, which was supported by atomistic modelling. Extensive molecular dynamics simulations by our cooperation partner T. Frolov revealed that for this specific tilt grain boundary shear stresses drive the phase transformation rather than temperature [3]. This is opposite to other ∑ [111] tilt GBs in Cu and Al, where we recently found that temperature can trigger the GB phase transformation.

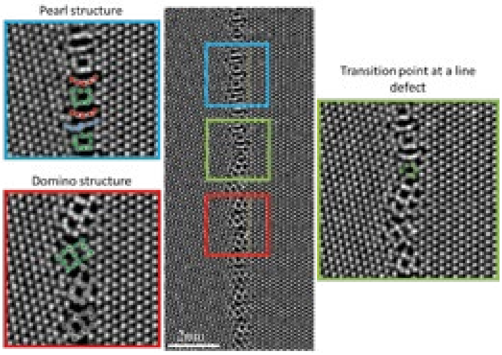

Fig. 1: *Scanning transmission electron microscope image of an asymmetric ∑19b [111] tilt GB in Cu consisting of pearl and domino structure separated by a disconnection.*

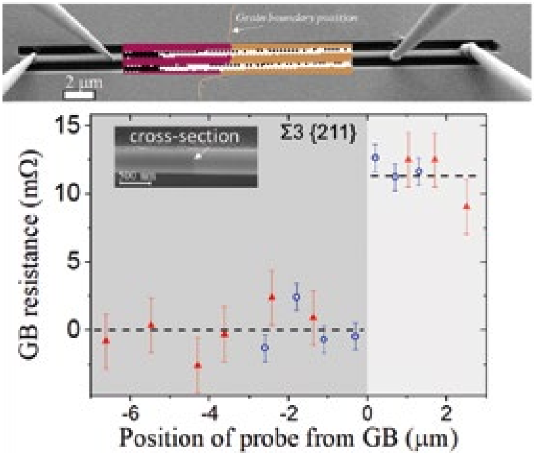

Fig. 2: *Four point in situ resistance measurement of a GB. Electron microscopy and diffraction is used to determine the GB character and position. A clear resistance signal is obtained for the incoherent* $\Sigma 3\ [111]\ (\bar{2}11)$ *GBs (red and blue data points).*

We also made a big leap forward in obtaining electrical properties from individual GBs. We developed a detailed methodology to probe *in situ* or *ex situ* the local resistivity of individual GBs [4]. For *in situ* measurements, electrical contacts were realized with W needles accurately positioned by piezo driven micromanipulators. GB resistivity follows the boundary excess volume and energy in a first approximation, but this correlation does not explain all resistivity-structure relations, indicating a contribution by the electronic structure of the boundary.

This project has received funding from the European Research Council (ERC) under the European Union's Horizon 2020 research and innovation programme (grant agreement No 787446).

References

1. Meiners, T.; Duarte, J.M.; Richter, G.; Dehm, G.; Liebscher, C.H.: Acta Mater. 190 (2020), 93.
2. Meiners, T.; Frolov, T.; Rudd, R.E.; Dehm, G.; Liebscher, C.H.: Nature 579 (7799) (2020) 375.
3. Bishara,H.; Ghidelli, M.; Dehm, G.: ACS Appl. Electron. Mater. 2 (7), (2020) 2049.

ERC-Consolidator Grant: Seeing hydrogen in metals

B. Gault

MA

This ERC-Consolidator project is called SHINE for Seeing Hydrogen IN mEtals. SHINE started in Feb. 2018 and is now well underway. We ambitioned to exploit the unique infrastructure of the Laplace Project [1] developed in the Atom Probe Tomography group at the MPIE to perform direct imaging and quantification of H atoms in candidate metallic alloys and materials for the hydrogen economy, to produce hydrogen by e.g. water splitting. This required the development of careful workflows to allow for electrochemical and, potentially, gaseous charging of hydrogen or deuterium under highly controlled conditions, and, subsequently, the transfer of the specimen to instruments enabling preparation and analysis. The resulting three-dimensional hydrogen mapping at the near-atomic scale was to be connected to physical properties and rationalized by correlating with atomistic simulations.

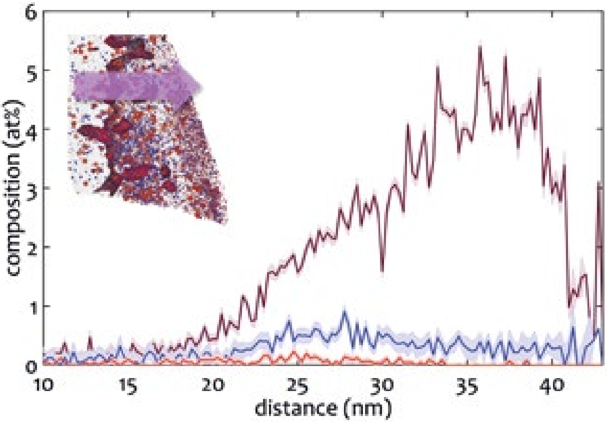

Fig. 1: *Distribution of D and H measured by APT at the interface between ferrite and decomposed cementite. [Ref. 3].*

The first three and a half years of the project have seen three postdoctoral scientists, including one based in the CM department, and two doctoral students work closely with a range of other colleagues from across the MPIE to push the frontiers of H-analysis by APT. We have: (1) demonstrated the cleanliness of cryo-enabled specimen preparation [1], in particular for hydride-forming materials, of cryo-transfer for air-sensitive materials [2]; (2) have discussed at length the details of the origins of the detected hydrogen detected within the APT specimen [3]; (3) we studied the field evaporation behaviour of hydrides and deuterides and estimated APT performance [4] and their growth mechanisms [5]; (4) we showcased the analysis of water and solutions, as well as liquid-solid interfaces [6].

Much more is to come and is under review, including via cross-departmental efforts. With experts from the GO department, we have developed the Reacthub Module (see p. 58) for gas-charging and processing of metallic alloys and oxides. We also synthesized catalysts nanoparticles with controlled doping levels and are testing their electrocatalytic activity. With the CM department on atomistic simulations to estimate the solubility of H in precipitates in 7XXX-Al alloys to explain the resistance to hydrogen embrittlement.

All these investigations have relevance to a ‘low-carbon-emission economy’ that humanity must develop in the 21st century, which was the framework in which SHINE was designed. Finally, a question came up during my interview for the grant in 2017 “what will be the legacy of SHINE?”, my answer was a better understanding of hydrogen’s behaviour in metals and the design of new, hydrogen resistant structural materials that can be fabricated on a large scale – and the recent joint work with Dirk Ponge’s group using chemical heterogeneity to enhance hydrogen resistance of high-strength steels appears like a perfect example [7].

This project has received funding from the European Research Council (ERC) under the European Union’s Horizon 2020 research and innovation programme (grant agreement No 771602).

References

1. Stephenson, L.T.; Szczepaniak, A.; Mouton, I.; Rusitzka, K.A.K.; Breen, A.J.; Tezins, U.; Sturm, A.; Vogel, D.; Chang, Y.; Kontis, P.; Rosenthal, A.; Shepard, J.D.; Maier, U.; Kelly, T.F.; Raabe, D.; Gault, B.: PLoS One 13 (2018) 1.
2. Chang, Y.; Lu, W.; Guénolé, J.; Stephenson, L. T.; Szczpaniak, A.; Kontis, P.; Ackerman, A. K.; Dear, F.; Mouton, I.; Zhong, X.; Raabe, D.; Gault, B.; Zhang, S.; Dye, D.; Liebscher, C. H.; Ponge, D.; Korte-Kerzel, S.; Raabe, D.; Gault, B.: Nat. Commun. 10 (2019) 942.
3. Breen, A. J.; Stephenson, L. T.; Sun, B.; Li, Y.; Kasian, O.; Raabe, D.; Herbig, M.; Gault, B.: Acta Mater. 188 (2020) 108.
4. Chang, Y. H.; Mouton, I.; Stephenson, L.; Ashton, M.; Zhang, G. K.; Szczpaniak, A.; Lu, W. J.; Ponge, D.; Raabe, D.; Gault, B.: New J. Phys. 21 (2019) 053025.
5. Mouton, I.; Chang, Y.; Chakraborty, P.; Wang, S.; Stephenson, L. T.; Ben Britton, T.; Gault, B.: Materialia 15 (2021) 101006.
6. El-Zoka, A. A.; Kim, S.-H.; Deville, S.; Newman, R. C.; Stephenson, L. T.; Gault, B.: Sci. Adv. 6 (2020) 49.
7. Sun, B.; Lu, W.; Gault, B.; Ding, R.; Makineni, S.K.; Wan, D.; Wu, C.-H.; Chen, H.; Ponge, D.; Raabe, D.: Nat. Mater. (2021).

ERC Starting Grant: Time-Scale bridging potentials for realistic molecular dynamics simulations

B. Grabowski

CM, now at University of Stuttgart, Materials Design

In 2015, B. Grabowski was awarded an ERC Starting Research Grant for his project proposal: *"TIME-BRIDGE: Time-scale bridging potentials for realistic molecular dynamics simulations"*. The project has been successfully finished in January 2021.

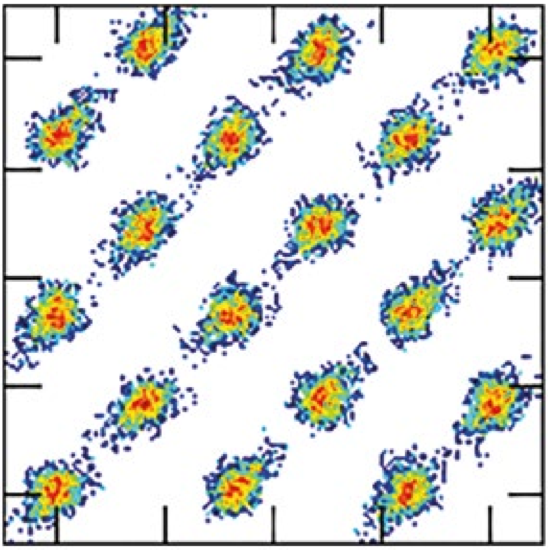

Fig. 1: *Two-dimensional histogram of ab initio molecular dynamics trajectories of a simulation for bcc Ti at 1025 K. The trajectories reveal a fingerprint of the martensitic transformation to the low-temperature hexagonal omega phase. Within TIME-BRIDGE new insights about this transformation have been revealed and have led to the discovery of a new class of structures, the linear-chain disordered structures [4].*

The superior aim of TIME-BRIDGE has been the design and application of novel methodologies that enable a more realistic atomistic simulation of materials and faithfully connect to experiment. This aim has been closely followed over the entire lifetime of the TIME-BRIDGE project. The specific efforts could be divided into three main areas of 1) bridging the time scale, 2) developing accurate interatomic potentials to reach larger system sizes than possible with *ab initio* methods, and 3) a close and faithful coupling of the simulations to nanoscale experiments. The over 40 published articles in renowned journals, 2 PhD theses, and 1 Master thesis demonstrate the excellent research output of TIME-BRIDGE. Three of the articles were published in Physical Review Letters, four in npj Computational Materials, ten articles in Physical Review B, and eight articles in Acta Materialia.

As for the time scale bridging, we have developed a novel strain reduction plus bond boost methodology to simulate thermally activated dislocation migration within the framework of accelerated molecular dynamics. With respect to interatomic potentials, we have combined machine-learning potentials with finite temperature *ab initio* simulations to efficiently obtain highly accurate materials properties. On the experimental side, we have performed extensive small-scale mechanical investigations that allowed us to analyze dislocation-grain boundary interaction. A breakthrough of TIME-BRIDGE was the novel methodology that combines ideas from the machine-learning field with concepts from finite temperature *ab initio* simulations and thereby enables high accuracy determination of materials properties. This method clearly advances the field beyond what was available before.

The project has had a significant impact on the career of the PI, B. Grabowski, and several team members. The PI has obtained a professor position at the University of Stuttgart and was awarded an ERC Consolidator Grant in 2019, Dr. Liam Huber became group leader at the MPIE, Dr. Prashanth Srinivasan was awarded the Humboldt fellow ship while working for the project, and several other members obtained high level positions in the academic field or industry.

This project has received funding from the European Research Council (ERC) under the European Union's Horizon 2020 research and innovation programme (grant agreement No 639211).

References

1. Ko, W.-S.; Choi, W. S.; Xu, G.; Choi, P.-P.; Ikeda, Y.; Grabowski, B.: Acta Mater. 202 (2021) 331.
2. Ko, W.-S.; Stukowski, A.; Hadian, R.; Nematollahi, A.; Jeon, J. B.; Choi, W. S.; Dehm, G.; Neugebauer, J.; Kirchlechner, C.; Grabowski, B.: Acta Mater. 197 (2020) 54.
3. Zhu, L.-F.; Körmann, F.; Ruban, A. V.; Neugebauer, J.; Grabowski, B.: Phys. Rev. B 101 (2020) 144108.
4. Korbmacher, D.; Glensk, A.; Duff, A. I.; Finnis, M. W.; Grabowski, B.; Neugebauer, J.: Phys. Rev. B 100 (2019) 104110.
5. Ikeda, Y.; Grabowski, B.; Körmann, F.: Mater. Charact. 147 (2019) 464.
6. Grabowski, B.; Ikeda, Y.; Srinivasan, P.; Körmann, F.; Freysoldt, C.; Duff, A. I.; Shapeev, A.; Neugebauer, J.: npj Comput. Mater. 5 (2019) 80.
7. Glensk, A.; Grabowski, B.; Hickel, T.; Neugebauer, J.; Neuhaus, J.; Hradil, K.; Petry, W.; Leitner, M.: Phys. Rev. Lett. 123 (2019) 235501.
8. Zhang, X.; Grabowski, B.; Körmann, F.; Ruban, A. V.; Gong, Y.; Reed, R. C.; Hickel, T.; Neugebauer, J.: Phys. Rev. B 98 (2018) 224106.

Humboldt Research Award: Grain growth in thin films – how microstructure depends on interfacial steps

D. Chatain

CNRS CINAM, Marseille, France

The Alexander von Humboldt Foundation awarded in November 2020 Dr. Dominique Chatain with the Humboldt Research Award in recognition of her outstanding academic record. Dr. Chatain will use the award to foster her interactions with the SN department and NG group at the MPIE with a focus on understanding microstructure evolution in metallic thin films. Advancing our current fundamental knowledge of the thermodynamic and kinetic parameters controlling grain growth in thin films is key to designing thin film microstructure. To address this topic, single crystalline α-Al_2O_3 substrates (sapphire) of different surface orientations are overgrown by a pure fcc metal or an alloy. These systems offer opportunities to diversely control the structural and chemical parameters to obtain a desired film microstructure spanning from specific textures to single-crystal films.

The very first point which is currently addressed is to understand the origin of the stable (st) orientation relationships (OR) of the grains of a pure Ni film on well-controlled sapphire substrates. Recent work of Chatain et al. [1] on fcc metallic films on fcc metal systems have shown that the OR is controlled by the substrate's surface steps, which emerge in a (slightly) miscut substrate due to surface energies. It is important to note that the direction of these steps can be different for the original substrate compared to the buried film/substrate interface. Thus, if two grain orientations of the film are originally present, one of them will be preferred because of the direction of the buried interfacial steps. Finally, it has been found by atomistic calculations [2] that during annealing interfacial diffusion is active above a certain temperature, which in turn can change the step direction and thus fosters the stability of a single orientation of the grains, which could eventually lead to single-crystal films by grain growth.

There are several interests of using sapphire as a substrate material: (1) it is an inert material avoiding reactions with many metals; (2) epi-polished surfaces of chosen orientation are easily available; (3) the trigonal sapphire structure (corundum) is more complex than the fcc structure of the metals investigated, and several kinds of well-known steps may appear on a single-crystal. We have focused on the c (0001)-plane of sapphire, which may have two possible kinds of steps running along crystallographically different perpendicular directions: $\langle 1\bar{1}00 \rangle$ and $\langle 11\bar{2}0 \rangle$. We will address the following questions: which orientation of these steps does stabilize at the interface with Ni, and how do these steps change at different temperatures.

First results on Ni and Cu films provide evidence on the influence of surface steps (Fig. 1). Depending on the original miscut of 0.25° in two perpendicular directions, a maze structure with two orientations and a nearly fully single crystalline structure emerge upon annealing under a hydrogenated atmosphere. Two different orientation relationships (OR1, OR2) have been grown on the c-plane of sapphire for several fcc metals (see e.g. [3]). However, why two or only one OR emerges is so far unclear.

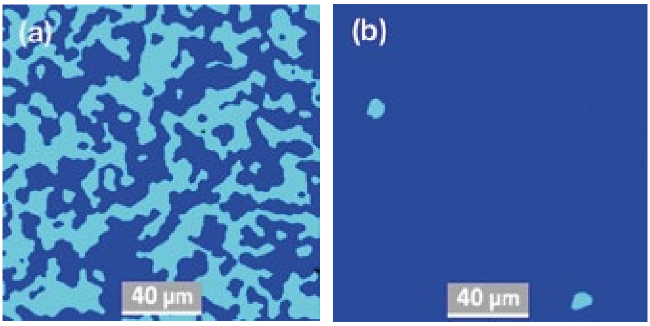

Fig. 1: *Preliminary results with Cu on sample with the two different miscut c-sapphires; EBSD maps of (a) a Cu maze <111> bi-crystal film on c-sapphire; (b) single-crystal with some residual "islands" of the second orientation. The two different blue colours correspond to the two twin-related grain orientations constituting this so-called "maze" bi-crystal.*

With our research we understand now why a maze structure (Fig. 1a) or a single crystal film evolves: the film is a maze bi-crystal of two twinned grains if the miscut is such that it reveals $\langle 1\bar{1}00 \rangle$ directions at sapphire surfaces. However, if the miscut reveals $\langle 11\bar{2}0 \rangle$ steps, the film quickly becomes a single-crystal.

To go further, there is a need to investigate the step direction at the interface after annealing. High resolution transmission electron microscopy observation of cross-sections of the samples will allow to prove how steps change at the interface between Ni and sapphire and explain the microstructure.

References

1. Wynblatt, P.; Chatain, D.; Rollett, A.D.; Dahmen, U.: Acta Mater. 168 (2019) 121.
2. Chatain, D.; Singh, S.; Courtois, B.; Silvent, J.; Verzeroli, E.; Degraef, M.: Rohrer, G.S.; Wynblatt, P.: Acta Mater. 200 (2020) 287.
3. Addab, Y.; Kini, M.K.; Courtois, B.; Savan, A.; Ludwig, A.; Bozzolo, N.; Scheu, C.; Dehm, G; Chatain, D.: Acta Mater. 200 (2020) 908.

Friedrich Wilhelm Bessel Research Award

Y. Zhu

Department of Mechanical and Aerospace Engineering, North Carolina State University, Raleigh, USA

The Friedrich Wilhelm Bessel Award of the Alexander von Humboldt Foundation is awarded to ~20 internationally renowned scientists each year. It is one of the most prestigious distinction for foreign scientists in Germany. In November 2020, Prof. Yong Zhu from the North Carolina State University (Raleigh, USA) was awarded the Friedrich Wilhelm Bessel Award for his outstanding achievements in nanomechanical testing of advanced materials. Prof. Zhu chose MPIE as his host institution and will work in close collaboration with the group of Dr. Christian Liebscher in the department SN to develop methodologies for observing atomic scale deformation processes in materials.

Prof. Zhu is a world leader in the field of mechanics of nanomaterials and stretchable electronics, including micro-electromechanical (MEMS) systems. He developed the first MEMS apparatus for mechanical testing of nanomaterials inside electron microscopes, representing a major milestone in the field. The MEMS-based device delivers incredibly small forces to be applied in a controlled manner to test even single nanowire geometries, while still being able to measure minute forces and observe the deformation processes.

Prof. Zhu has performed pioneering experiments utilizing the micromechanical apparatus that have led to a mechanistic understanding how size and surface effects impact the mechanical properties of nanomaterials. He and his team have unraveled the effect of hydrogen embrittlement in metallic nanowires at the nanoscale. Prof. Zhu and collaborators could demonstrate by a combination of *in situ* TEM nanomechanical testing and atomistic simulations that a transition in failure mechanism occurs from distributed plasticity to localized necking in metallic nanowires due to the presence of surface-adsorbed hydrogen [1]. In another piece of work, Prof. Zhu and co-workers showed by *in situ* testing in the TEM of bi-twinned nanowires that the twin boundary promotes two competing deformation mechanisms, namely dislocation slip and delocalized plasticity via anomalous tensile detwinning [2].

A major limitation to observe atomic scale deformation processes is imposed by instabilities in the commercial mi- cro- and nanomechanical testing devices. However, Prof. Zhu's unique lab-on-a-chip testing platform will enable the observation of how metallic alloys deform at atomic resolution and elevated temperatures. This tool is one-of-a kind and the MEMS-based apparatus is currently employed and tested in the *in situ* holder platforms available at the SN department.

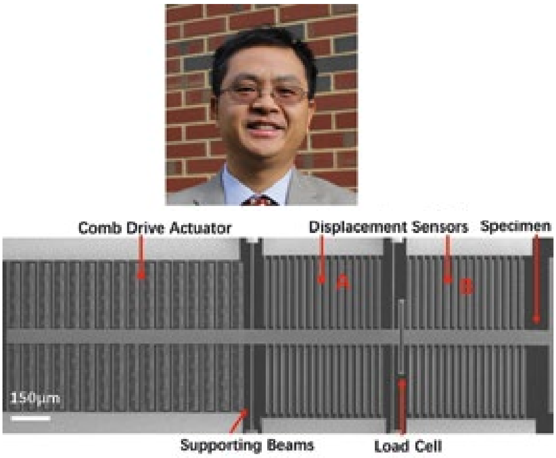

Fig. 1: *Picture of Prof. Yong Zhu and the MEMS-bases nanomechanical testing apparatus he developed. This system is utilized in the in situ heating and biasing holder of MPIE to probe atomic scale deformation processes in aberration-corrected TEMs.*

The synergies in Prof. Zhu's outstanding expertise in nanomechanics and the capacity at MPIE in material development and characterization offer a rich playground for a fruitful scientific interaction. Together, we will tackle to resolve the underlying atomistic mechanisms responsible for hydrogen embrittlement in twinning induced plasticity steels, while focusing to probe one of the most critical microstructural components, twins, by *in situ* probing. We further aim to explore the atomic scale deformation behavior in novel high entropy alloys at temperatures of up to 500 °C. This also requires a novel design of the MEMS-based nanomechanical testing device. One of our goals is to resolve deformation processes at grain boundaries and how dislocations interact with them at atomic resolution. The nanomechanical testing device will elevate our capabilities to even study the impact of grain boundary phase transformations and segregation on their plastic behavior. In a final step, it is planned that we test tellurium nanowires inside of our aberration-corrected TEMs to observe stress induced phase decomposition in these 1D nanomaterials.

References

1. Yin, S.; Cheng, G.; Chang, T.H.; Richter, G., Zhu, Y.; Gao, H.: Nat. Commun. 1 (2019) 2004.
2. Cheng, G.; Yin, S.; Chang, T.H.; Gao, H.; Zhu, Y.: Phys. Rev. Let. 119 (2017) 256101.

Heisenberg-Professorship: Metallic composite materials

H. Springer

MA, now also at: Institute of Metal Forming, RWTH Aachen University, Germany

In October 2019, H. Springer joined the RWTH Aachen University as professor after successfully applying for the Heisenberg program of the German Research Foundation (DFG), and now heads the academic and research department for metallic composite materials. After an apprenticeship as an industrial mechanic, studying mechanical engineering and working as a welding engineer in the automotive industry, H. Springer joined the MPIE as a PhD student in 2008. He subsequently led the group combinatorial metallurgy and processing from 2011 onwards, and completed his habilitation at the RWTH Aachen University in 2018.

Fig. 1: *Main research areas of the Heisenberg professorship for metallic composite materials: From self-healing structural materials to novel materials for lightweight design.*

The Heisenberg professorship is concerned with the design and development of innovative metallic composite materials. The vast spectrum of constituents concerning their morphology, microstructure, mechanical, physical and chemical properties offers huge potential to create materials capable of shifting technological frontiers. The following topic areas are at the core of ongoing research at both the RWTH and MPIE (Fig. 1): High modulus steels offer the possibility to overcome the inherent conflict between the key properties for lightweight design, namely strength, density and stiffness. Based on previous research into the alloy concepts [1], the focus now lies on their maturation towards industrial application. This is directly linked to the development of *in situ* nano-composites using newly emerging processing technology (Fig. 2), especially additive manufacturing [2,3]. Bulk composites with constituents on the meso- to macroscale, on the other hand, are not only relevant for new applications in energy conversion, but can also be utilised as model materials to systematically study and optimise the effects of inhomogeneities or interfaces on the bulk property profile. Another target is to incorporate the capacity for self-healing as a new functionality to increase lifespan of structural materials.

The research on these topics follows an efficient and holistic approach based on both scientific and industrial experience, with three key components to be further broadened and developed: Acceleration via combinatorial methods [4], property driven iterations and integrated design by early incorporation of application-critical parameters and utilisation of conventionally unwanted phenomena for new material solutions.

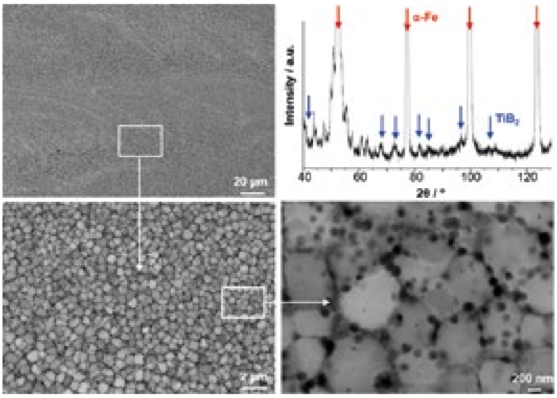

Fig. 2: *Ideal match between novel material concepts and synthesis technologies: in situ nano structuring of high modulus steels during additive manufacturing [3].*

The integration of the Heisenberg professorship with the continued research activities within the new group for "Sustainable Material Science and Technology" (see p. 29) and the interdepartmental group "Materials Science of Additive Manufacturing" (see p. 28) further strengthens the collaboration with the RWTH Aachen University.

References

1. Springer, H.; Aparicio-Fernandez, R.; Duarte, M.J.; Kostka, A.; Raabe, D: Acta Mater. 96 (2015) 47.
2. Springer, H.; Baron, C.; Szczepaniak, A.; Jägle, E.A.; Wilms, M.B.; Weisheit, A.; Raabe, D.: Mater. Des. 111 (2016) 60.
3. Springer, H.; Baron, C.; Mostaghimi, F.; Poveleit, J.; Mädler, L.; Uhlenwinkel, V: Addit. Manuf. 32 (2020) 101033.
4. Baron, C.; Springer, H.; Raabe, D.: Mater. Des. 112 (2016) 131.

VIDI Award:
How to mix the perfect high entropy alloy cocktail?

F. Körmann

CM

Since 2018 the Dutch Research Organisation (NWO) supports the research of Dr. Fritz Körmann, project group leader in the Computational Materials Design department and researcher at the Delft University of Technology in the Netherlands with the prestigious five-years group funding VIDI award. The VIDI award enables researchers to establish a research group in the Netherlands and to develop their own line of research. The research of Körmann is about compositionally complex alloys including the class of concentrated solid solutions alias high entropy alloys.

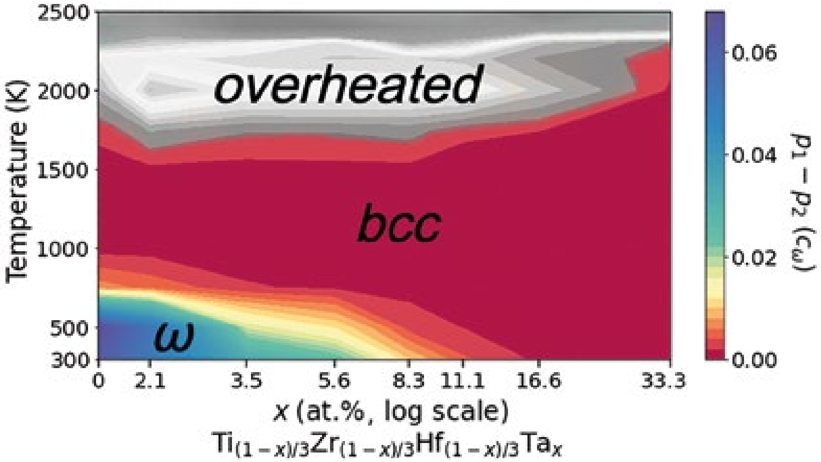

Fig. 1: *Application of developed active learning machine-trained potentials to explore the phase stability of TiZrHfTa$_x$ depending on Ta concentration x and temperature [5]. The different structural phases, i.e., omega and BCC, can be clearly distinguished based on a developed structural descriptor [5] (colour coded).*

Compositionally complex alloys (CCAs) and high entropy alloys (HEAs) have received great attention in terms of the underlying physics responsible for their formation as well as unusual physical and materials properties. Whereas traditional alloys usually contain 1-2 main components, CCAs are comprised of typically 3-6 elements in high or even equal concentrations and crystalize into surprisingly simple lattice structures with randomly dispersed atomic species. Due to the gigantic chemical configuration space, computational techniques [1] are critical to narrow down promising compositions.

Within the last years several methodological developments have been achieved to overcome the underlying computational and simulation limits for such complex alloys. In particular techniques based on novel machine-learning based interatomic potentials were developed allowing, e.g., to compute vibrational free energies of multicomponent alloys with hitherto unachievable accuracy [2]. Highly efficient on-lattice machine learning potentials were employed to explore the phase stability and short-range order in a number of multicomponent alloys [3].

In multicomponent body-centred cubic (BCC) alloys, the temperature-dependent intrinsic phase stability between omega, bcc and hcp affects crucially the mechanical properties. Novel concepts have been developed to distinguish between omega and bcc phases [4]. Further developed machine-learning potentials combined with active learning concepts enabled to screen for the phase stability of these alloys in a fully automatic fashion [5].

References

1. Ikeda, Y; Grabowski, B.; Körmann, F.: Mater. Charact. 147 (2019) 464.
2. Grabowski, B.; Ikeda, Y.; Srinivasan, P.; Körmann, F.; Freysoldt, C.; Duff, A.; Shapeev, A.; Neugebauer, J.: npj Comput. Mater. 5 (2019) 80.
3. Kostiuchenko, T.; Körmann, F.; Neugebauer, J.; Shapeev, A.: npj Comput. Mater. 5 (2019) 55.
4. Ikeda, Y.; Gubaev, K.; Neugebauer, J.; Grabowski, B.; Körmann, F.: npj Comput. Mater. 7 (2021) 34.
5. Gubaev, K; Ikeda, Y.; Tasnádi, F.; Neugebauer, J.; Shapeev, A.; Grabowski, B.; Körmann, F.: Phys. Rev. Mat. 5 (2021) 073801.
6. Oh, H.S.; Kim, S.J.; Odbadrakh, K.; Ryu, W.H.; Yoon, K.N.; Mu, S.; Körmann, F.; Ikeda, Y.; Tasan, C.C.; Raabe, D.; Egami, T.; Park, E.S.: Nat. Comm. 10 (2019) 2090.

Pushing the boundaries of temperature and strain rate for *in situ* nano- and micromechanical testing

J.P. Best, M.J. Duarte Correa, R. Ramachandramoorthy

SN

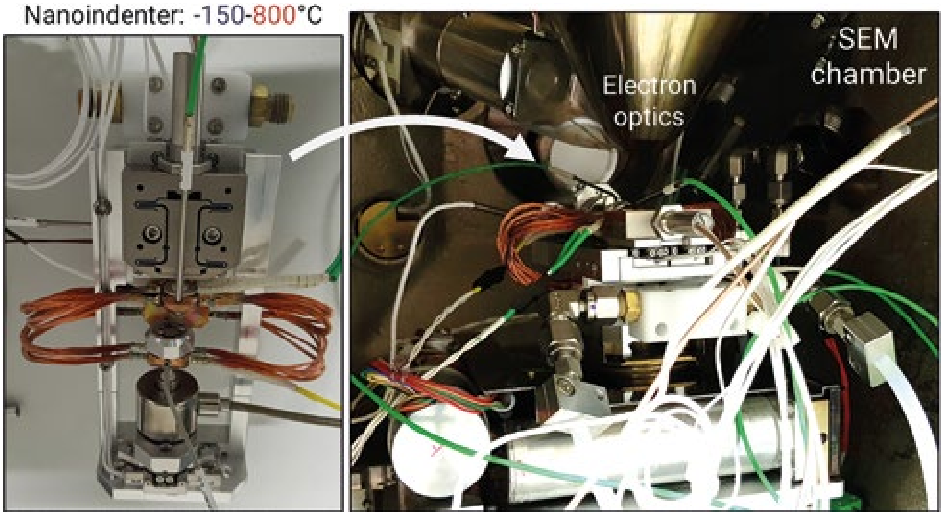

Fig. 1: *The nanoindentation system set-up for cryo temperature and quasi-static indentation of fused quartz within a tungsten-filament SEM. The modular system can be modified for heating, cooling or high-speed measurements and the requisite water-cooling tubes and cabling fed-through the uniquely designed ports on the SEM. Alterna-tively, the system can be mounted in a field-emission gun-based SEM or an environmental chamber for measurements with either high resolu-tion imaging or under controlled atmospheres for performing cutting-edge hydrogen-loading experiments.*

The most cutting-edge nano- and micromechanical materials research look to perform *in situ* measurements, such as inside a scanning electron microscope (SEM), at variable temperatures and testing speeds (strain rates). This allows for research investigations of either the controlling material deformation mechanisms, or properties (such as the strength, elastic modulus or fracture toughness) under application relevant conditions. Such characterizations are critical for furthering current research into, amongst others, grain boundary plasticity in metals, hydrogen loading into advanced engineering materials, and brittle-to-ductile toughness measurements of intermetallics and thin films.

The newly acquired nanoindentation system allows for performing *in situ* SEM nano- and micromechanical testing and is unique in a number of critical ways: (i) capability for testing at high temperatures (800 °C) and cryogenic temperatures (-120 °C); (ii) true displacement control (as opposed to load control), which is required for stable fracture and crack-growth measurements; (iii) versatility to be fitted with electrochemical cells and used under extreme environments such as in the presence of hydrogen (and other gasses if needed); (iv) ability to perform tests and acquire data at very high strain rates (~10^3-10^4 s^{-1}) to understand time-dependent deformation mechanisms. This combination of functions renders the newly commissioned system unique for small-scale mechanical testing under extreme loading conditions.

The instrument is currently shared between three research groups in the SN department, led by J. Best, M. J. Duarte Correa, and R. Ramachandramoorthy, to perform a suite of advanced nano- and micromechanical *in situ* testing. For hydrogen embrittlement experiments in Duarte's group, the modular system will be installed inside an environmental chamber with the capability to introduce nitrogen to have an oxygen-free atmosphere. Hydrogen is provided by electrochemical charging by using a custom-made electrochemical cell similar to one already developed in the group. For variable temperature, strain rate and microscale toughness measurements in Best's group, two separate SEMs for *in situ* mechanical measurements can accommodate the system. Ramachandramoorthy's group will primarily focus on using the system for extreme *in situ* micro/nanomechanical testing at combinations of high strain rates and high/cryo temperatures, while also developing protocols/instrumentation to push the dynamic limits of the system even further.

Alemnis AG is acknowledged for its support in the installation and commissioning of the device.

Large-volume 3D microstructure characterization using EBSD and mechanical serial polishing

S. Zaefferer[1], P. Konijnenberg[2], T. A. Griffiths[3], S.-P. Tsai[1]

[1]MA, [2]now at: Forschungszentrum Jülich , [3]now at: University of Vienna

Understanding materials properties based on the materials microstructure is one of the key tasks of materials science. All properties of materials are influenced by the 3-dimensional nature of materials. The corrosion resistance of stainless steels, for example, depends on the 3D structure of grain and phase boundaries, the efficiency of polycrystalline solar cells is influenced by the 3D arrangement of grain boundaries, and the strain hardening behaviour of aluminium alloys depends on the 3D arrangement of dislocations and on the shape and arrangement of precipitations.

A number of tools for 3D microstructure characterization exists but it is obvious, that there is a need for a technique bridging between the high-resolution and the large-volume techniques, i.e. bridging between microstructures and macroscopic properties. This gap is closed with new large-volume 3D electron backscatter diffraction (EBSD) measurement techniques based on broad ion beam (IB) sputtering, plasma-focussed IB sputtering, laser ablation or mechano-chemical polishing. We believe that the latter has the greatest potential for the following reasons:

- It delivers very large, perfectly flat and defect free surfaces, also on multi-phase materials.
- The parameters of polishing can be modified very widely to cope with very different materials.
- Removal rates can be widely changed and controlled by selection of polishing parameters.
- It is mechanically robust and keeps the microscope in perfectly proper conditions.

Following earlier work of other researchers, we developed, over the last three years, a system for large volume 3D EBSD microstructure analysis, which is now fully functional (Fig. 1). The core of the system is a polishing automaton modified from a commercially available device. This instrument allows fully automated polishing of samples with a large number of well-controlled parameters. It, furthermore, contains a cleaning and drying station such that a completely clean and dry sample is delivered finally. Sample exchange is done via a self-built transfer station inside of the machine and a collaborative robot arm. A scanning electron microscope runs in a fully automated manner to perform large-area EBSD measurements: The measurement of the removal thickness occurs by observing cross sections of markers that have been placed, using FIB, on a side of the sample during sample preparation. The whole system runs fully automated, controlled by a master computer, which orchestrates all individual devices via self-written software. A few numbers illustrate the power of the system:

- Targeted measurable volume: up to 500 x 500 x 500 µm³ with a 1 µm step size in every dimension.
- Measurement rate: about 30 slices per day with maps 500 x 500 pixels.
- Largest measured amount of data points per 3D measurement: around 30.000.000.

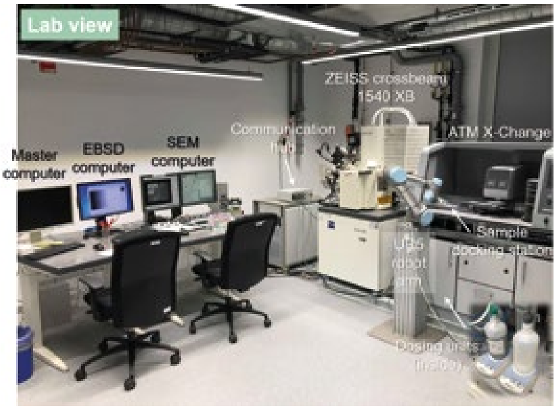

Fig. 1: *Overview on the large-volume 3D EBSD setup based on chemo-mechanical serial sectioning.*

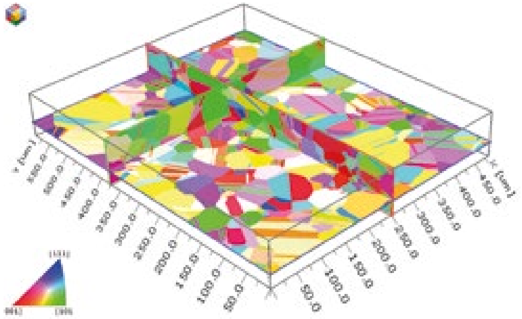

Fig. 2: *3D EBSD data set of a 316 stainless steel, obtained with the large volume 3D EBSD system. The microstructure is displayed and analysed with the program QUBE, programmed by P. Konijnenberg.*

Fig. 2 shows one example of a 3D data set, collected from a 316 stainless steel sample in order to obtain a statistically reliable set of grain boundaries for the study of grain boundary corrosion. The data set contains about 600 grains and a few thousands grain boundaries, including twin boundaries. After the 3D measurement, the remaining sample was submitted to a corrosive medium and the corrosion behaviour of the grain boundaries, whose characteristics are known from the 3D experiment, was recorded and analysed. This work is ongoing.

ReactHub module: towards *in situ* atom probe tomography

L.T. Stephenson[1], H. Khanchandani[1], S.-H. Kim[1], A. El-Zoka[1], U. Tezins[1], A. Sturm[1], D. Vogel[2], B. Gault[1]

[1]MA, [2]GO

Since 2017, the MPIE has been actively developing a world-unique suite of instruments interconnected by a set of ultra-high-vacuum (UHV) suitcases with the capability to transport specimens at cryogenic temperatures. This suite is under the umbrella of the Laplace Project [1] and includes a glovebox, a scanning-electron microscope – focused ion beam (SEM-FIB) and two atom probes [1]. A new gas-reaction chamber called the ReactHub Module (RHM) is now also online and was added to the Laplace suite over the course of 2020.

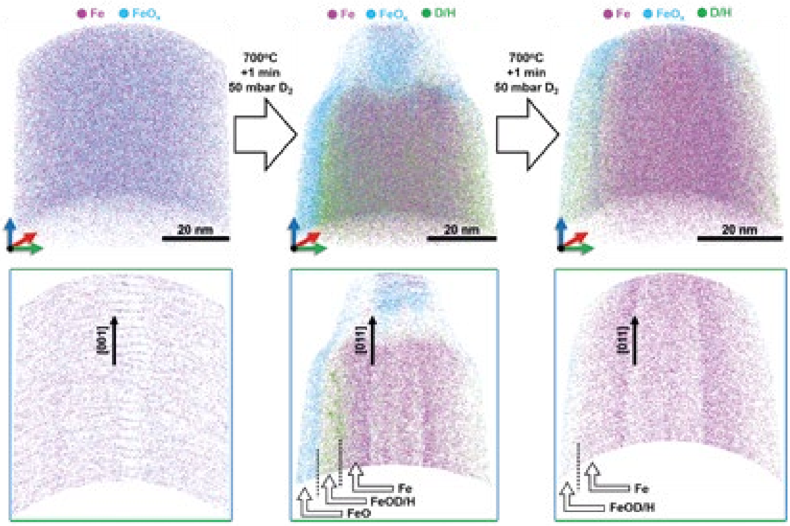

Fig. 1: *Transient state captured during the direct-hydrogen reduction of FeO with the RHM.*

In the past decades, approaches have been used to study processes 'as they take place' *in situ* in a microscope, e.g. while the specimen is heated, deformed or a chemical reaction is taking place. Atom probe tomography (APT) being a destructive analysis technique, *in situ* approaches cannot directly be performed or implemented. Some have considered the end of the needle-shaped APT specimen as an individual nanoparticle, and the technique has a long-history of studying catalytically-active surfaces by performing surface reactions with gases, including at high-temperature [2], but only after the reaction has taken place. Field electron emission microscopy has sometimes been combined to follow the reaction *in situ*.

In the RHM, an individual specimen can be heated up to approx. 1000 ˚C by targeting it with a laser. The chamber is initially ultrahigh vacuum, but pressures of up to 1 bar of either O_2, N_2, or H_2 can be introduced. There is an additional line for a gas bottle that can be changed whenever necessary. One of the key interests is that the specimen is sitting on a cryogenically-cooled stage (< 50 K) so as to quench extremely fast. By kinetically freezing the specimen in its reactive state, we expect to get closer to performing *in situ* observations of transient chemical states. Following quenching, the specimen is taken into the pre-cooled cryo-UHV suitcase and transported into the SEM-FIB for further preparation or the atom probe for analysis. The SEM-FIB is equipped with a cryo-stage so as to perform cryogenic specimen preparation [3].

We showcase here two *quasi-insitu* studies. We started investigating the influence of the microstructure on the direct reduction of Fe-oxides by hydrogen, in order to understand the influence of the microstructure on the limiting rate of the reduction reaction [4]. Fig. 1 showcases the direct reduction of a Fe-oxide single crystal prepared in the form of atom probe needles performed using the RHM. After 1 minute of reduction, the reaction front can be visualized and its composition precisely measured to inform models of the reaction kinetics. Some more evolution is found after a second minute of reduction. The QR code will take you to a video of the complete workflow from the atom probe to the RHM.

References

1. Stephenson, L. T.; Szczepaniak, A.; Mouton, I.; Rusitzka, K. A. K.; Breen, A. J.; Tezins, U.; Sturm, A.; Vogel, D.; Chang, Y.; Kontis, P.; Rosenthal, A.; Shepard, J. D.; Maier, U.; Kelly, T. F.; Raabe, D.; Gault, B.: PLoS One 13 (2018) 1.
2. Barroo, C.; Lambeets, S. V.; Devred, F.; Chau, T. D.; Kruse, N.; De Decker, Y.; Visart De Bocarmé, T.: New J. Chem. 38 (2014) 2090.
3. Chang, Y.; Lu, W.; Guénolé, J.; Stephenson, L. T.; Szczpaniak, A.; Kontis, P.; Ackerman, A. K.; Dear, F.; Mouton, I.; Zhong, X.; Zhang, S.; Dye, D.; Liebscher, C. H.; Ponge, D.; Korte-Kerzel, S.; Raabe, D.; Gault, B.: Nat. Commun. 10 (2019) 942.
4. Kim, S.-H.; Zhang, X.; Ma, Y.; Souza Filho, I. R.; Schweinar, K.; Angenendt, K.; Vogel, D.; Stephenson, L. T.; El-Zoka, A. A.; Mianroodi, J. R.; Rohwerder, M.; Gault, B.; Raabe, D.: Acta Mater. 212 (2021) 116933.

The hydrogen lab

M. Rohwerder, A. Vogel, D. Vogel

GO

From hydrogen embrittlement of high strength steels or aluminium alloys to hydrogen production and storage, the effect of hydrogen on the physical and chemical properties of materials is an important topic of current research. For this reason, it is not surprising that hydrogen in materials is one of the key research topics at the MPIE, where all four departments have strong activities, often in close cooperation between the departments.

Especially quantitative detection of hydrogen in materials is important for providing a reliable basis for fundamental investigations of hydrogen embrittlement and hydrogen-related corrosion phenomena. Setting a sound basis for research already starts with realising a reproducible and well controlled hydrogen loading into the materials of interest. The easiest way to reach high hydrogen activities at the surface and thus also high concentrations in the material, is to use electrochemical hydrogen loading, often with the help of so-called recombination inhibitors. However, the efficiency of this loading, i.e. the achieved hydrogen activity and its constancy throughout the loading process, are depending crucially on the investigated material, its surface preparation and the applied electrolyte and recombination inhibitor. Principally it is necessary for each new material to be investigated to elaborate a reliable and well controlled loading routine, which usually requires significant experience in electrochemistry. Even more challenging is carrying out hydrogen permeation studies by the so-called Devanathan-Stachurski method. Since in the GO department novel approaches were developed in the recent years, especially based on the Kelvin probe technique, it was decided to establish a central lab, the "Hydrogen Lab", in order to make the different techniques available at the MPIE accessible for researchers, partially also as a service, especially for the more sophisticated ones. The most important techniques offered are various electrochemical and also gas-phase based hydrogen loading set-ups, Thermal desorption analysis (TDA) and hydrogen permeation set-ups. TDA has long been used in characterizing different hydrogen traps inside materials. At the MPIE significant experience was gained over the last years with a self-built TDA set-up, but an easier to use system was needed for broader use and is now available in this Hydrogen Lab.

Furthermore, the GO department is working on the development of a novel TDA system, which will be constructed around a specially developed mass spectrometer and an infrared heater. However, in TDA measurements, the diffusible hydrogen (hydrogen at interstitial sites and shallow traps) is often not detected, as e.g for ferritic steels it effuses usually before the measurement can be started. The Davanathan-Starchurski permeation technique can cover this shortage and also provide further information, such as e.g. on the effective diffusion constant. However, for such experiments to provide reliable and exact results, the entry side and especially the exit side of the material to be investigated usually have to be coated by thin palladium layers. Especially at the exit side, which has to be polarised to sufficiently high enough potentials to ensure the complete oxidation of all permeating hydrogen, the stability of the palladium layer is an important issue. In order to avoid oxidation of the underlying substrate the palladium layer needs to be pore-free and well adhering. This can be a challenge and even impossible to achieve for some materials. The Kelvin Probe based approach developed at the MPIE (see p. 68) solves this problem, as it just requires an ultra-thin Pd PVD layer, which does not need to adhere too well or to be fully pin-hole free, and is easy to apply on different materials [1]. However, significant expertise is necessary for achieving reliable results. Hence, as part of the lab Kelvin Probe based permeation techniques will be made available by an experienced team.

Fig. 1: *View through the Hiden TPD Workstation Viewport © Hiden Analytical Ltd.*

Besides different set-ups for hydrogen charging, the TDA, melt extraction, and the Kelvin Probe based permeation techniques, also set-ups for electrochemical sample thinning will be provided. This is a key prerequisite for enabling permeation measurements to be carried out on samples with ultra-low permeability.

In summary, a number of different techniques related to providing information on hydrogen in materials are made available to all researchers working on related topics at the MPIE.

References

1. Wu, C. H.; Krieger, W.; Rohwerder, M.: Sci. Technol. Adv. Mat. 20 (2019) 1073.

Nano-IR spectroscopy by AFM-IR

M. Rabe, P. Ebbinghaus

GO

For the study of local material surface properties, such as surface chemistry and conformation, the combination of microscopic and vibrational spectroscopic information yields valuable information. However, the lateral resolution of classical vibrational microspectroscopy is limited to a few µm due to the Abbe limit, which permits the study of nanometric structures. One method that circumvents this limitation consists of a coupling of atomic force microscopy with IR spectroscopy (AFM-IR). It employs the near field between AFM tip and sample, created by light incident at a shallow angle from wavelength tuneable IR laser sources (Fig. 1).

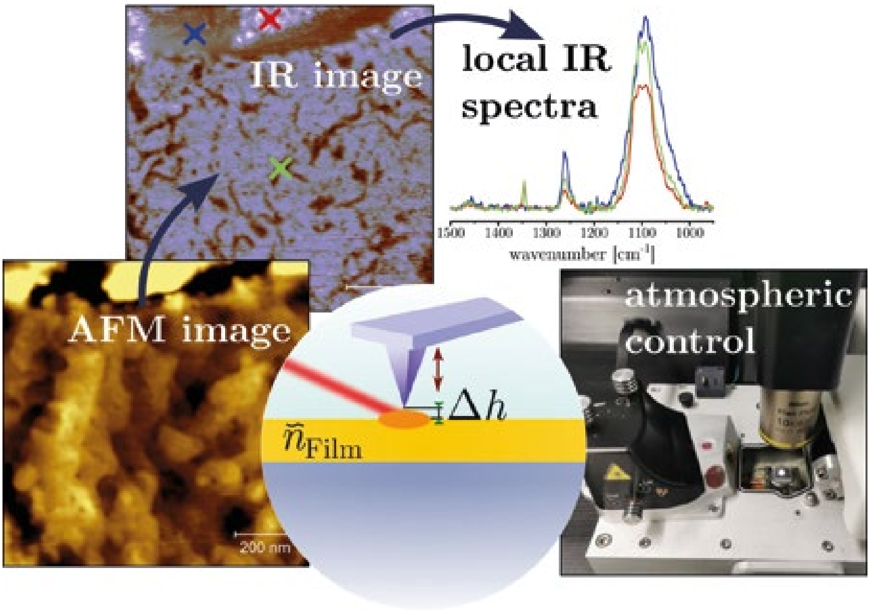

Fig. 1: *Illustration of the measuring principles of photothermal AFM-IR and photo of open sample chamber with piezo stage and measurement head. AFM and spectral data obtained from an organic monolayer of oligo-ethyleneglycol-terminated thiols on gold (111). The IR intensity image measured at 1347 cm^{-1} reveals local density and/or conformational variations. IR-imaging and local IR spectroscopy with lateral resolutions down to 10 nm can be achieved.*

The new AFM-IR setup was acquired in 2020. It uses the photothermal detection mode, in which the thermal expansion of the sample upon IR absorption is detected by the AFM cantilever. This principle allows a direct measurement of the sample absorption coefficient, i.e. spectra directly comparable to transmission spectra can be obtained without modelling. For an increased sensitivity a resonance enhancement (RE) is achieved by using pulsed sources with their frequency tuned to the cantilever oscillation. In this way RE AFM-IR can yield spectroscopic information with lateral resolutions down to ~10 nm and reaches monolayer sensitivity.

Because the method is based on the conventional AFM technique, the acquired nanometric spectral information can be correlated to sample topography and other nano-mechanical properties that can be obtained from the coincident AFM measurements. For being applicable to a wide variety of hard and soft samples, the new system allows nano-IR spectroscopy in contact or tapping mode, where the latter features reduced tip-sample interactions. Different modes allow acquisition of point spectra, spectral images and hyperspectral images.

Two laser sources are available: a 4-chip quantum cascade laser that spans the wavenumber region of 950 to 1900 cm^{-1} and a new generation OPO laser that covers the region of 2700 to 3900 cm^{-1}. The now reachable region around 3500 cm^{-1} is specifically interesting for the planned study of properties of ad- or absorbed water whose IR stretching modes appear in this region and contain valuable information on the water hydrogen bonding network. For such studies the system is uniquely equipped with a mechanism that allows the transfer of samples under controlled atmosphere conditions and an environmental control chamber that allows atmospheric and humidity control up to 95 %rh. Furthermore, the linear polarisation of the incident laser light can be varied with respect to the plane of incidence, which allows for polarimetric studies of anisotropic materials.

Magnetic materials

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Magnetic materials enable the electrification of transport, communication, energy, and manufacturing. They serve for instance as hard magnets in electrical motors or as soft magnets in transformers. Their remanence, coercivity, and hysteresis losses determine the efficiency of devices that are urgently needed for enabling society and economy to use power from sustainable energy sources.

Several aspects make magnetic materials attractive study objects for the institute, in its transition towards a microstructure- and systems-oriented research facility, as many of their properties derive from an interplay of processing, microstructure, and chemistry, at scales that reach from manufacturing dimensions (e.g. Goss texture formation in secondary recrystallization) down to the electronic scale (e.g. defects in Heusler alloys). This turns research on magnetic materials not only a multi-physics and multi-scale problem, but requires also close collaboration between characterization, processing, and theory, disciplines where the institute has strength. The MPIE joined forces in this field with the groups of O. Gutfleisch at TU Darmstadt, R. E. Dunin-Borkowski at the Ruska Center for high-performance electron microscopy in Jülich, and with M. Acet and M. Farle at the University Duisburg-Essen. The activities include bi-weekly seminars and fuel the collaborative research center CRC/TRR 270 'Hysteresis design of magnetic materials for efficient energy conversion' (see p. 94).

We work with soft and hard magnetic materials as well as invar alloys. Soft ferromagnetic materials are alloys that magnetise and demagnetise at low hysteresis losses. We study particularly Fe - 3.2 mass % Si steels [1], which serve in electrical power transformers, motors, and generators. The Si increases the electrical resistivity, reducing eddy current losses. Grain-oriented Fe-Si steel used for non-rotating applications, i.e. transformers, has a strong preferred crystallographic orientation. In Fe the easiest directions of magnetisation are the <001> crystal directions. In grain-oriented Fe-Si steel the Goss orientation, i.e. the {110}<001> orientation, is realised through abnormal grain coarsening to minimise magnetic losses. Our research deals with the mechanisms behind the abnormal growth of crystals with this specific orientation. While earlier approaches suggested inhomogeneous solute and particle pinning as possible causes, our recent results reveal a dislocation substructure in the grain interiors. These seem to promote the local dissolution of second phase pinning centres, thus affecting the grain's growth behaviour.

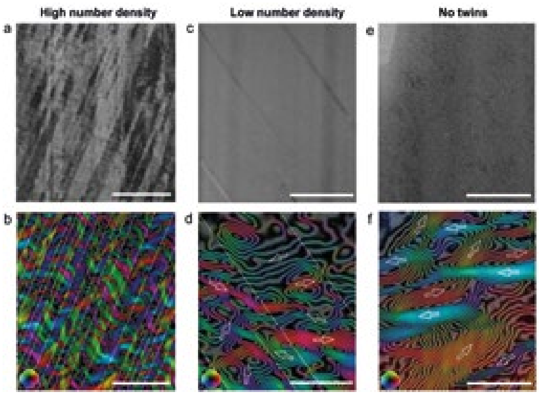

Fig. 1: *Magnetic domain structure of τ-MnAl with high- and with low-densities of micro-twins. (a, c, e) TEM images and (b, d, f) the corresponding in-plane magnetic induction map of magnetic domains extracted from off-axis electron holography.*

Another project line is about rare-earth-free magnets, particularly Heusler phases and high entropy alloys. Regarding binary Heusler alloys we study the τ phase variant of the $Mn_{55}Al_{45}$ alloy. This is a metastable phase, which forms from the high temperature hexagonal-close packed (hcp) ε-phase when suppressing the competing equilibrium β-Mn (cubic) and γ_2 (Al_8Mn_5, rhombohedral) phases. The equilibrium phases are non-magnetic and their presence, even in trace fractions in the alloy, deteriorates the magnetic properties. During growth, the τ phase forms twins, antiphase boundaries, and dislocations, due to growth misplacements at the interphase boundary. This defect structure influences the magnetic hysteresis of the ferromagnetic τ phase alloy, where specifically twins act as domain wall nucleation and pinning sites. We could show that twinning involves significant segregation of Mn along the twin boundaries by combining transmission electron microscopy (TEM) and atom probe tomography (APT). An enrichment of ~8 at.% Mn was measured along the twin boundaries with a confined depletion outside of it, suggesting short range diffusion occurring during massive transformation. This finding shows that the design of effective domain wall pinning centres in such materials needs to consider both, their structural and their confined chemical decoration state, Fig. 1 [2, 3].

References

1. Frommert, M.; Zobrist, C.; Lahn, L.; Böttcher, A.; Raabe, D.; Zaefferer, S.: J. Magn. Magn. Mater. 320 (2008) 657.
2. Palanisamy, D.; Raabe, D.; Gault, B.: Scr. Mater. 155 (2018) 144.
3. Palanisamy, D.; Raabe, D.; Gault, B.: Acta Mater. 174 (2019) 227.

Model processes for hydrogen plasma and direct reduction of iron ore

H. Springer

MA, now also at: Institute of Metal Forming, RWTH Aachen University, Germany

Decarbonisation of the steel production to a hydrogen-based metallurgy is one of the key steps towards a sustainable economy. While still at the beginning of this transformation process, with multiple possible processing routes on different technological readiness, we conduct research into the related fundamental scientific questions at the MPIE. At the core of these efforts is the development of suitable experimental methodologies to elucidate the underlying physical, chemical, and microstructural phenomena governing the reduction kinetics and boundary conditions. Currently, two processes for the sustainable reduction of iron ore – as the most critical step in a carbon-neutral steel production – are being developed and deployed.

Hydrogen-based direct reduction (HyDR) is a major contender for the future of green ironmaking without a direct release of CO_2 emissions. It relies on a multistep solid-gas reaction, where iron ore in pellet or powder form is subjected to elevated temperatures under a reducing gas atmosphere. The fundamental understanding of such a complex reaction process is necessary from the thermodynamics and kinetics perspectives. The in-house designed thermo-gravimetric analysis (TGA) setup coupled with mass spectrometry at the MPIE ([1]) allows us to precisely measure the reduction kinetics of iron oxides under well-defined gaseous atmospheres (containing e.g. H_2, NH_3, or CO). The infrared furnace enables extremely fast and well-defined ramping up to 10 K/s and a high operational temperature up to 1000 °C. During the HyDR experiments, the *in situ* weight change of the iron oxides can be continuously tracked by a sensitive thermo-balance with an accuracy of 0.1 µg to quantify the reduction degree (i.e. loss of oxygen) [2]. Furthermore, the gas composition near the sample surface can be analysed using a fused capillary attached to the quadrupole mass spectrometer. Such an additional technique provides valuable information on the alternation of the gas species during HyDR, indicating the reaction paths and kinetics. With the expansion of the research activities in hydrogen-based metallurgical science and technology at the MPIE, new equipment, such as a TGA setup with the capability of mixing different gases and a fluidised-bed setup is under construction. This development will allow for in-depth investigations and further optimisation of complex industrial processes.

However, the fragmented sponge iron coming out of the HyDR process needs to be molten in order to be processed into various steel alloys. This is typically performed in an electric arc furnace (EAF), resulting in a major energy penalty. That motivates the hydro plasma smelting reduction (HPSR) process. Here the iron ore is molten and simultaneously reduced in an EAF with a hydrogen plasma arc, offering thermodynamic and kinetic advantages, but on a yet lower technological readiness level [3]. We study the fundamental aspects of the HPSR process, for example regarding ore composition and plasma parameters, by use of a modified, lab-scale, arc-melting furnace (Fig. 1). While being smaller and less complex than an industrial EAF, it allows us to systematically study the phenomena in the most relevant zone, i.e. where the hydrogen containing plasma interacts with the molten and partially ionised iron oxides. Currently ongoing is the implementation of improved diagnostic devices such as mass spectroscopy of the reactor atmosphere and spectral analysis of the plasma constituents, together with academic and industrial partners, within the group of “Sustainable Material Science and Technology” (see p. 29).

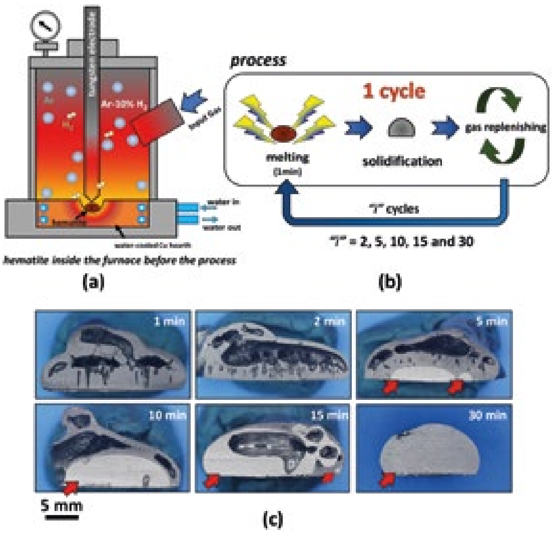

Fig. 1: *Experimental model process for the hydrogen plasma smelting reduction [3]: (a) modified arc-melter, (b) scheme of kinetic experiments, and (c) cross section showing the progressing reduction of liquid iron ore.*

References

1. Auinger, M.; Vogel, D.; Vogel, A.; Spiegel, M.; Rohwerder, M.: Rev. Sci. Instrum. 84 (2013) 085108.
2. Kim, S.-H.; Zhang, X.; Schweinar, K.; Souza Filho, I.; Angenendt, K.; Ma, Y.; Vogel, D.; Stephenson, L.; El-Zoka, A.; Mianroodi, J.; Rohwerder, M.; Gault, B.; Raabe, D.: Acta Mater. 212 (2021) 116933.
3. Souza Filho, I.R.; Ma., Y.; Kulse, M.; Ponge, D.; Gault., B.; Springer, H.; Raabe, D.: Acta Mater 213 (2021) 11697.

Challenges of ECCI on light metals

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MA

Electron channelling contrast imaging (ECCI) is a powerful technique for observation of extended crystal lattice defects (e.g. dislocations, stacking faults) with almost transmission electron microscopy (TEM) like appearance but on bulk samples in the scanning electron microscope (SEM). ECCI has been proven to give good results on materials that have an average atomic number larger than approx. 20 (calcium). Titanium, steels, Ni-base alloys, tungsten, pyrite (FeS_2), CdTe, and many other metals and compounds give very good channelling contrast. In contrast, for the lighter materials, e.g. silicon, aluminium, magnesium and many minerals, e.g. quartz, the contrast is significantly worse and achieving proper channelling is challenging. The reasons for this are manifold: the most important is the lower electron scattering power of the lighter elements, but lower crystal symmetry and preparation artefacts, among others, also play a role.

Due to the lower scattering power the primary electrons penetrate deeper into the material. As a result the electron wave “senses“ the atom channels of the crystal to a larger depth below the surface, which means that the beam may have a higher probability to encounter atom columns, bent due to the presence of elastic strains. As a consequence, an original channelling situation (allowing visualization of lattices defects) may turn into a backscattering one (which veils lattice defects). This happens particularly on ductile metal, and may strongly reduce the channelling contrast and visibility of defects. Even slightly deformed light metals may, therefore, not show any useful channelling contrast.

The most efficient way to handle the lower scattering power is to reduce the acceleration voltage. This increases contrast and allows in many cases to observe good channelling contrast on light metals. Fig. 1 displays an example of a small angle grain boundary in a 7075 aluminium alloy, solution annealed and slightly aged, imaged at 20 kV (instead of the usual 30 kV). An electro-polishing procedure at low temperatures (-30 °C) excluding water allowed proper ECCI preparation of the material.

The case of magnesium is even more challenging than that of aluminium. First, Mg creates an even thicker oxide film than Al-Zn alloys which prevents from “seeing through it” with low energy electrons. Final preparation needs to be done by extended ion polishing. A second, and equally important reason is the hexagonal closed packed (hcp) crystal structure. Compared to the face centred cubic (fcc) structure, the diffraction intensities are much reduced and less homogeneously distributed over reciprocal space. In fact, the strongest reflector in the hcp structure is the (0001) plane of which only one exists. In the cubic structure the {111} planes (4) and the {011} planes (6) all allow good imaging. This fact is displayed in fig. 2, showing simulated electron channelling patterns for Al and Mg. Nevertheless, if preparation is optimum and the strongest reflectors are used for imaging, dislocation structures may be visualized very well also in Mg alloys. An example is shown in fig. 3 displaying the <c+a>-type dislocations that form inside of extension twins. And also here the advantages of ECCI over TEM, namely observation of large bulk samples and performance of *in situ* experiments, may be highly valuable.

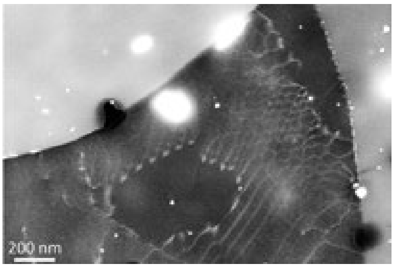

Fig. 1: *ECCI of a small angle grain boundary in a 7075 Al alloy, imaged at 20 kV. The large angle grain boundary at the right is decorated with newly precipitated particles. of a Zn-rich phase.*

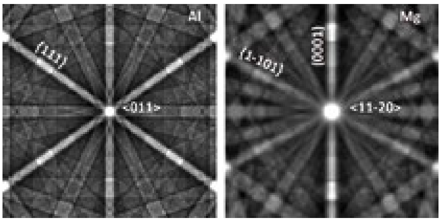

Fig. 2: *ECC patterns simulated with the software EMSoft by Marc de Graef, for fcc Al (atomic number Z=13) and hcp Mg (Z=12). Al shows much stronger, sharper and more homogeneously distributed reflectors.*

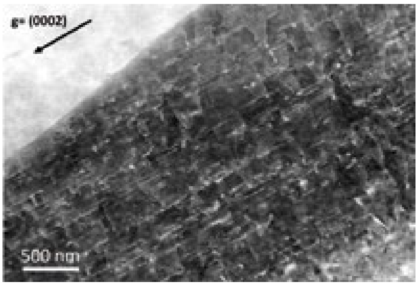

Fig. 3: *ECCI of dislocations in an extension twin of a slightly deformed Mg-Ca-Zn alloy. Imaging with g=(0002) makes <c+a> dislocations visible.*

Additively micromanufactured fluid-filled metal microvessels: APT sample carriers

E. Woods[1], S. Kang[2], L. Stephenson[1], B. Gault[1], R. Ramachandramoorthy[2]

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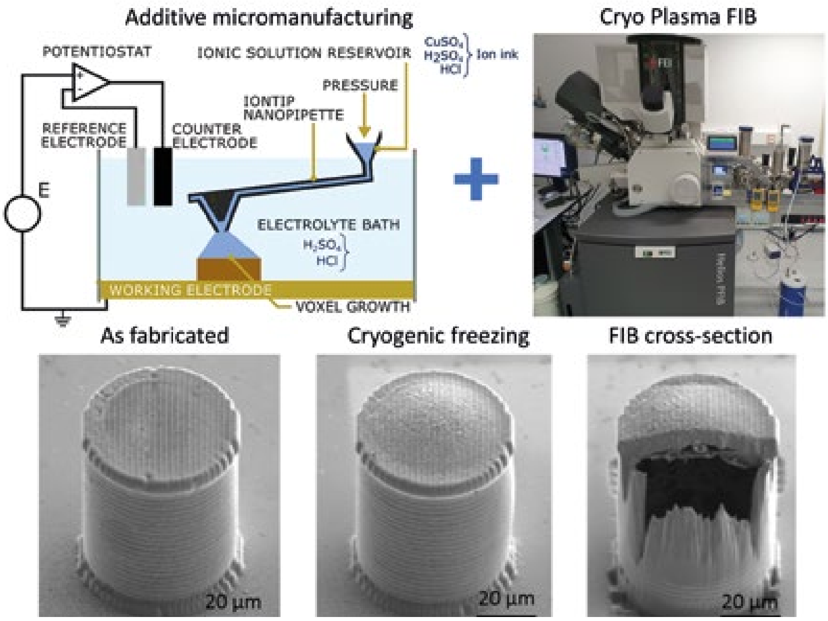

Fig. 1: *Top: Process flow involving additive micromanufacturing followed by cryogenic milling using a PFIB. Bottom: A copper micropillar with encapsulated water, which was subsequently frozen resulting in bulging side and top walls – seen more clearly from FIB cross-section.*

Atom probe tomography (APT) is a material analysis technique capable of 3D compositional mapping with sub-nanometer resolution. The specimens for APT are shaped as sharp needles (~100 nm radius at the apex), so as to reach the necessary intense electrostatic fields, and are typically prepared via focused ion beam (FIB) based milling. Though well-established protocols exist for analyzing solid materials using APT, studying wet chemical systems such as nanoparticle dispersions is still a new frontier owing the inherent challenges of preparing and transporting liquid media under cryogenic conditions (20-80 K) [1]. To enable such studies on frozen liquids containing small metallic or biological objects, the development of specimen preparation approaches that provide a stable storage platform for such liquid media is required. In other words, the challenge is to identify a manufacturing method capable of encapsulating liquid media with different dispersants inside a stable microscale metal container, which can be further frozen and shaped using cryo FIB into a needle suitable for APT [2].

Using a recently developed additive micromanufacturing (AMM) method, we are developing a unique synthesis platform to fabricate fluid-filled metal microvessels. The additive micromanufacturing method uses localized electrodeposition of individual sub-micron scale metal droplets to create complex 3D architectures (Fig. 1). This manufacturing process is electrodeposition-based and done submersed inside a supporting conductive liquid. This provides a unique pathway to fabricate metal microvessels encapsulated with the supporting liquid bath, which can be tuned for pH and dispersants such as metal/biological nanoparticles.

The process flow steps we envisage are 3D microprinting of various geometries whereby micron-scale vessels are deposited onto a typical support for atom probe specimens (coupons, grids), then locally filled with the liquid that contains the material of interest, e.g. nanoparticles, organics, proteins etc. Following controlled freezing, the vessel will then be turned into a needle-shaped specimen suitable for APT by using a cryo-plasma FIB (PFIB). Recently, a proof-of-concept study whereby copper microvessels with encapsulated water was fabricated using AMM printer (Fig. 1).

References

1. El-Zoka, A. A.; Kim, S. H., Deville, S.; Newman, R. C.; Stephenson, L. T.; Gault, B.: Sci. Adv. 6 (2020) 49.
2. Gault, B.; Chiaramonti, A.; Cojocaru-Mirédin, O.; Stender, P.; Dubosq, R.; Freysoldt, C.; Makineni, S.K.; Li, T.; Moody, M.; Cairney, J.M.: Nat. Rev. Methods Primers. 1 (2021) 1.

Advanced electron microscopy techniques: *in situ* SEM/TEM and 4D-STEM

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H. Bishara[1], J. Best[1], G. Dehm[1]*

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Electron microscopes offer unique capabilities to probe materials with extremely high spatial resolution. Recent advancements in *in situ* platforms and electron detectors have opened novel pathways to explore local properties and the dynamic behaviour of materials. Within the scanning electron microscope (SEM), *in situ* mechanical measurements allow for investigation under variable testing conditions such as temperature and strain rate on a wide range of geometries fabricated using focused ion beam (FIB) milling. We develop novel microscale fracture testing geometries for *in situ* observation of crack growth. Further, a robust *in situ* SEM methodology has been recently designed for electrical measurements with high spatial resolution [1] and high sensitivity allowing for resistivity measurement of individual microstructural features such as grain boundary segments. Using the method, we successfully analyzed the resistivity-structure relations of grain boundaries and dislocations in Cu, thermoelectric materials [2] and metal oxides, while on-going research targets the effects of chemical segregation on local resistivity.

Aberration-corrected transmission electron microscopes (TEMs) enable the exploration of complex materials at atomic resolution. We develop novel sample preparation strategies, data acquisition schemes and algorithms to enable *in situ* heating observations down to atomic resolution. Our goal is to quantify the dynamic microstructure evolution of materials at elevated temperatures. We utilize micro-electro-mechanical systems (MEMS) based heating chips (Fig. 1) with ultra-high stability and temperature control. This system was used to explore novel precipitation pathways of nano-carbides in nano-twinned regions of interstitially alloyed high entropy alloys [3]. We further optimized sample geometries to explore their phase decomposition at 900 °C and atomic resolution (Fig.1). The fast acquisition of high resolution data enables us to devise new algorithms to quantify the dissolution dynamics of the nano-carbides. Thermoelectric materials harvest energy from a temperature gradient, but the microstructures at the hot end are rarely characterized. Our recent study reveals that the dislocation networks designed to scatter phonons in PbTe can be stabilized at higher temperatures by pinning Cottrell atmospheres [4]. As another example, we observed Ostwald ripening in nanostructured oxides, which was beneficial to their photocatalytic activity [5].

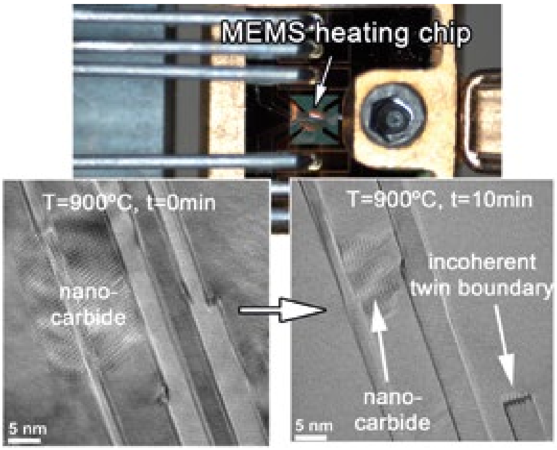

Fig 1: *Top: Picture of the in situ heating holder with electrical contacts and the MEMS-chip, where the FIB-prepared sample is mounted to. Bottom: Two snapshot images of an atomic resolution heating sequence captured at 900°C showing the dissolution of a nano-carbide and associated de-twinning process in an interstitially alloyed high entropy alloy.*

The advancement of fast electron detectors not only enables the dynamic observation of materials under *in situ* conditions with high time resolution, but also renders the development of novel imaging modalities possible. We advanced precession electron diffraction assisted 4D-STEM techniques, where a full electron diffraction pattern is captured at every beam position in a scanning TEM (STEM). These large-scale datasets provide rich crystallographic information of samples at nanometer resolution for extracting local grain and phase orientations in nanomaterials [6]. Ultimately, we strive to combine *in situ* probing with 4D-STEM to establish a complete quantitative picture of microstructure evolution.

References

1. Bishara, H.; Ghidelli, M.; Dehm, G.: ACS Appl. Electron. Mater. 2 (7) (2020) 2049.
2. Luo, T.; Serrano-Sánchez, F.; Bishara, H.; Zhang, S; Villoro, R.B.; Kuo, J.J.; Felser, C.; Scheu, C.; Snyder G.J.; Best, J.P.; Dehm, G.; Yu, Y.; Raabe, D.; Fu, C.; Gault, B.: Acta Mater. 217 (2021) 117147.
3. Lu, W.; Liebscher, C.H.; Yan, F.; Fang, X.; Li, L.; Li, J.; Guo, W.; Dehm, G.; Raabe, D.; Li, Z.: Acta Mater. 185 (2020) 218.
4. Abdellaoui, L.; Chen, Z.; Yu, Y.; Luo, T.; Hanus, R.; Schwarz, T.; Bueno Villoro, R.; Cojocaru-Miredin, O.; Snyder, G.J.; Raabe, D.; Pei, Y.; Scheu, C.; Zhang, S.: Adv. Funct. Mater. 31 (2021) 2101214.
5. Zhang, S.; Diehl, L.; Wrede, S.; Lotsch, B.V.; Scheu, C.: ***Catalysts*** ***10*** (2020) 13.
6. Jiwon, J.; Cautaerts, N.; Dehm, G.; Liebscher, C.: Microsc. Microanal. (2021) 1.

Environmental small-scale mechanics

M.J. Duarte Correa, L. Bierbrodt, R. Ramachandramoorthy, S.G. Kang, J.P. Best

SN

The field of micromechanics has seen a large progress in the past two decades, enabled by the development of instrumented nanoindentation. Consequently, diverse methodologies have been tested to extract fundamental properties of materials related to their plastic and elastic behaviour and fracture toughness. Established experimental protocols are carried out either at atmospheric conditions or at ultra-high vacuum inside an electron microscope. Our aim at the MPIE is to expand the methodology to either minimize the influence of varying experimental and instrumental factors (such as atmospheric humidity, temperature, and oxygen content) or to mimic service conditions.

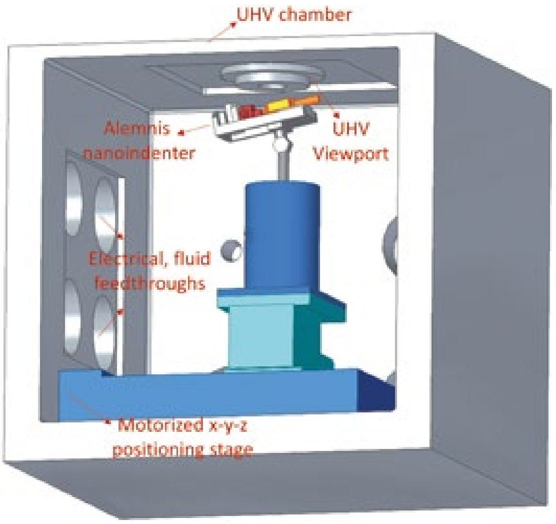

Fig. 1: *CAD design of the environmental chamber for conducting micromechanical experiments under ultra-high vacuum (UHV) or different atmospheres, mimicking real-life working conditions.*

A custom-made environmental chamber was built to host a micromechanical testing device. The steel chamber can operate in vacuum UHV up to 10^{-6} mbar or in different gas atmospheres such as inert gas (nitrogen or argon), forming gas or dry air. In the original design a high-temperature microscale loading rig (also tailor-made) was installed in the chamber for micromechanical testing at temperatures up to 600 °C in vacuum [1]. Current adaptations to the chamber allow hosting a newly acquired nanoindentation system. Tension, compression, bending and fatigue tests can be performed in this true displacement-controlled system in standard and extreme conditions, like variable temperature (-120 to 800 °C) or high strain rates (up to ~10^3-10^4 s^{-1}). In addition, this modular system can accommodate our custom-designed electrochemical cell for hydrogen charging and extreme conditions [2].

This system, composed by the nanoindenter adapted inside the environmental chamber, is currently shared between three newly established groups in the SN department working on micromechanics in different environments:

- The group of M. J. Duarte Correa studies the deformation processes and fracture behaviour of materials under different chemical/electrochemical environments, and particularly during hydrogen charging. A customized electrochemical cell designed in-house [2] and adapted to the modular nanoindenter system will be used to understand the mechanisms leading to hydrogen embrittlement. The gas atmosphere provided in the chamber will serve to minimize hydrogen desorption and formation of concentration gradients.
- R. Ramachandramoorthy's group will primarily use the environmental chamber to conduct dynamic testing of micro-to-meso scale architectures under application-relevant atmospheric conditions including high-humidity (for biomaterials) and inert gas (such as argon for high/cryo temperature testing). Specifically, using a combination of optical microscope and high-speed camera the deformation processes during high strain rate mechanical testing of complex 3D architectures will be captured.
- The group of J. Best looks to investigate controlled microscale crack growth and plasticity of engineering alloys, along with variable temperature measurements, under different environmental conditions such as local oxidative or inert gas (such as argon for high/cryo temperature testing).

References

1. Arigela, V.G.; Palukuri, N.R.; Singh, D.; Kolli, S.K.; Jayaganthan, R.; Chekhonin, P.; Scharnweber, J.; Skrotzki, W.: J. Alloys Compd. 790 (2019) 917.
2. Duarte, M.J.; Fang, X.; Rao, J.; Krieger, W.; Brinckmann, S.; Dehm, G.: J. Mater. Sci. 56 (2021) 8732.

Illuminated scanning flow cell – operando study on light-induced power generation & degradation

S. Zhang[1], A.M. Mingers[2], O. Kasian[2], S.-H. Kim[3], B. Gault[3], C. Scheu[1]

[1]NG, [2]GO, [3]MA

Photovoltaic materials have seen rapid development in the past decades, propelling the global transition towards a sustainable and CO_2-free economy. Storing the day-time energy for night-time usage has become a major challenge to integrate sizeable solar farms into the electrical grid. Developing technologies to convert solar energy directly into hydrogen fuels would not only ease grid operation, but also power other energy demands, e.g. fuel cell vehicles.

Current photovoltaic materials have excellent conversion efficiency, but they need to be shielded from the environment, precluding their use in water electrolysis. A new class of materials, photoelectrodes, is being developed which combines both functions of harvesting solar energy and converting them into hydrogen by electrolysis. As photoelectrodes are witnessing ever increasing power conversion efficiency, their lifetime has become the limiting factor for their economic return, making it pivotal to understand how these materials degrade under operation conditions.

To address this specifically, we have built up an illuminated scanning flow cell (iSFC) system to enable operando measurements, as plotted in Fig. 1. The electrolyte is confined in the flow cell to reach defined surface areas of the photoelectrode. With monochromated or broad-band illumination coupled to potentio- or galvano-static control, the photo-activity of the materials can be evaluated. At the same time, the outflow of the electrolyte is analysed by a mass spectrometer to quantify the photo-corrosion *in operando*.

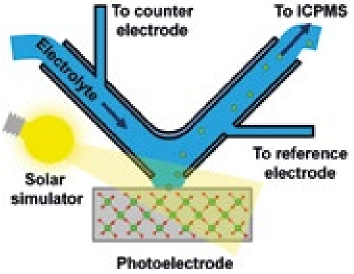

Fig. 1: *Schematic of the illuminated scanning flow cell (iSFC) setup coupled to inductively coupled plasma mass-spectrometry (ICPMS).*

So far, we have applied iSFC measurements to quantify the dissolution of WO_3 [1] and $BiVO_4$ [2,3] photoanodes during operation, which have been considered stable since decades. We revealed that while stable in the dark, they can rapidly degrade once the illumination introduces enough minority carriers on their surface. The surface chemistry also plays a major role, as vastly different photo-corrosion rates are measured in various electrolytes [3].

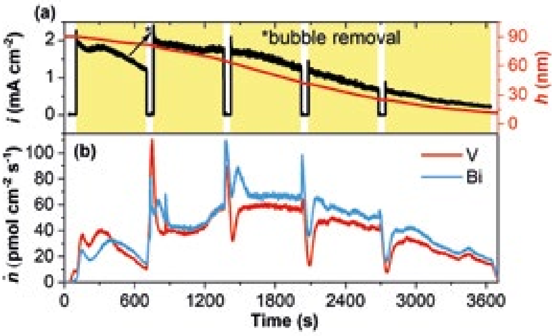

Fig. 2: *A life cycle study on a $BiVO_4$ photoanode [3]. (a) Photocurrent density i (black), film thickness h (red), and (b) dissolution rates of V and Bi from $BiVO_4$ under 1.6 V in borate buffer electrolyte. Windows with 100 mW cm^{-2} illumination are highlighted in yellow.*

Before our operando measurements, many considered a photoelectrode stable by showing a stable photocurrent with time. We have refuted this assumption in our recent work [3] using a life cycle study of $BiVO_4$ (Fig. 2). The photocurrent did not decrease until the $BiVO_4$ film degraded to less than ~60 nm, the diffusion length of the minority carrier to yield the photocurrent. We used this example to alert the community that photocurrent stability on thicker photoelectrodes cannot prove photostability, whereas independent photo-corrosion measurements are essential.

Having demonstrated the limited stability for some of the best photoanodes, they still have a bright future for application. Within the community, we are developing catalytic layers on photoanodes that simultaneously improve photo-activity and stability. To unveil the critical junctions and give them lifetime assessment, we are fostering local synergy at the MPIE to correlate the operando iSFC measurements with surface characterisation at the GO department, atom probe tomography at the MA department, and electron microscopy at the NG group.

References

1. Knöppel, J.; Zhang, S.; Speck, F.D.; Mayrhofer, K.J.J.; Scheu, C.; Cherevko, S.: Electrochem. Commun. 96 (2018) 53.
2. Zhang, S.; Rohloff, M.; Kasian O.; Mingers, A.M.; Mayrhofer, K.J.J.; Fischer, A.; Scheu, C.; Cherevko, S.: J. Phys. Chem. C 123 (2019) 23410.
3. Zhang, S.; Ahmet, I.; Kim, S.-H.; Kasian O.; Mingers, A.M.; Schnell, P.; Kölbach, M.; Lim, J.; Fischer, A.; Mayrhofer, K.J.J.; Cherevko, S.; Gault, B.; Van de Krol, R.; Scheu, C.: ACS Appl. Energy Mater. 3 (2020) 9523.

Scanning Kelvin Probe for advanced measurement of hydrogen and electrochemical activity at buried interfaces

M. Rohwerder, C.-H. Wu, M. Prabhakar

GO

The utilization of Kelvin Probe (KP) techniques for spatially resolved high sensitivity measurement of hydrogen has been a major break-through for our work on hydrogen in materials. A relatively straight forward approach was hydrogen mapping for supporting research on hydrogen embrittlement that was successfully applied on different materials, and this cooperation was continued, see e.g. [1, 2].

The key strength of this KP approach for H mapping is that the hydrogen release from the investigated material into an ultra-thin palladium detection layer is directly measured with high local resolution. This release rate is a measure for how fast H can be supplied e.g. to a crack tip. Hence, this measured H activity is more relevant than just the local concentration of hydrogen without information of how strongly it is trapped. However, information related to different local distribution of hydrogen activity can only be resolved if the release of features at the surface is larger than the global background release from the bulk. For materials with very fine microstructure which have been loaded with hydrogen to a depth much larger than the average length scale of this microstructure, local differences of the hydrogen release rate are averaged out as long as the hydrogen activity in the loaded region is still relatively high.

To solve this problem is one main focus for the further development of SKP- force microscopy (SKPFM) for mapping hydrogen activity and permeation. In order to be able to identify local differences and especially also trap sites with moderate and even low release rates of the trapped hydrogen, this global background has to be significantly lowered. One possible approach that is investigated is ultra-short hydrogen loading prior to the measurements, another one, which is currently favoured, is the thinning-out of the materials to ultra-thin slabs, thus reducing hydrogen supply from the bulk. A procedure for this is currently elaborated for use on various materials and will be made available in the recently established Hydrogen Lab (see p. 59).

Since often the evaporation of a thin Pd film and providing a dry nitrogen atmosphere is inconvenient or even impossible (such as in the field), alternatives are currently being investigated. Hence, another important activity is trying to provide an understanding of the potential distribution measured on the surface of not Pd coated materials. Depending on the H activity in the material the potential of the surface oxides can be either directly correlated to an electrochemical equilibrium of the oxygen reduction reaction (ORR) and the hydrogen oxidation reaction (HOR) on the surface of the oxide, just as it was observed on the surface of Pd (see e.g. [3]), or it may just be correlated to the oxide forming in the presence of high hydrogen activity directly after termination of the hydrogen loading procedure. This will depend on the kind of surface oxide as well as on the hydrogen activity prevailing directly upon termination of loading and is object of intense current research.

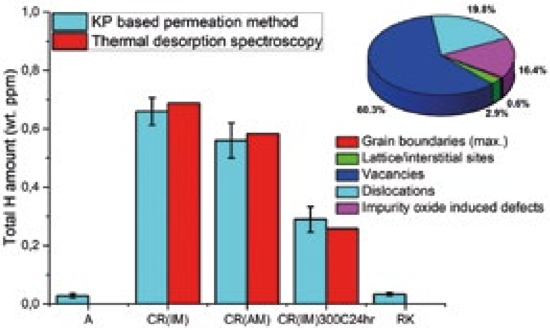

Fig. 1: *A comparison of total hydrogen amount in annealed (A), cold-rolled (CR(IM: induction melted); CR(AM: arc melted)) or re-crystallized (RK) 5 wt.% Ni ferritic steels, obtained by the hydrogen electrode-based method and by thermo-desorption analysis All samples with same thickness (1 mm) were loaded with hydrogen under same conditions (*a_H*: 60). Insert: distribution for CR.*

Another application, rather of SKP than SKPFM, is for substituting the standard Devanathan-Stachurski approach for permeation measurements. A huge advantage of the Kelvin Probe based approach is that it is sufficient to evaporate an ultra-thin Pd film on the exit side, which is much easier to achieve than the usually required electrodeposition of a well-adhering and pore-free palladium coating. As could be quite impressively demonstrated the KP-based permeation method can indeed provide fully quantitative information (Fig.1).

This approach is made available in the Hydrogen Lab (see p. 59). Current research on this application is focussed now on realizing Kelvin Probe based permeation measurements also at different temperatures.

References

1. Krieger, W.; Merzlikin, S.V.; Bashir, A.; Szczepaniak, A.; Springer, H.; Rohwerder, M.: Acta Mater. 144 (2018) 235.
2. Sun, B.H.; Krieger, W.; Rohwerder, M.; Ponge, D.; Raabe, D.: Acta Mater. 183 (2020) 313.
3. Zhong, X.K.; Schulz, M.; Wu, C.H.; Rabe, M.; Erbe, A.; Rohwerder, M.: Chemelectrochem. 8 (2021) 712.
4. Wu, C.H.; Krieger, W.; Rohwerder, M.: Sci. Technol. Adv. Mat. 20 (2019) 1073.

Providing *ab initio* simulation techniques to describe the dynamics and reactions at electrified interfaces

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[1]CM, [2]GO

Developing and providing accurate simulation techniques to explore and predict structural properties and chemical reactions at electrified surfaces and interfaces is critical to surmount materials-related challenges in the context of sustainability, energy conversion and storage. The groups of C. Freysoldt, M. Todorova and S. Wippermann develop various methods to incorporate finite electric fields in density-functional theory (DFT) and apply them to answer fundamental questions in corrosion, field evaporation, and the thermodynamics and transformation of electrochemical interfaces.

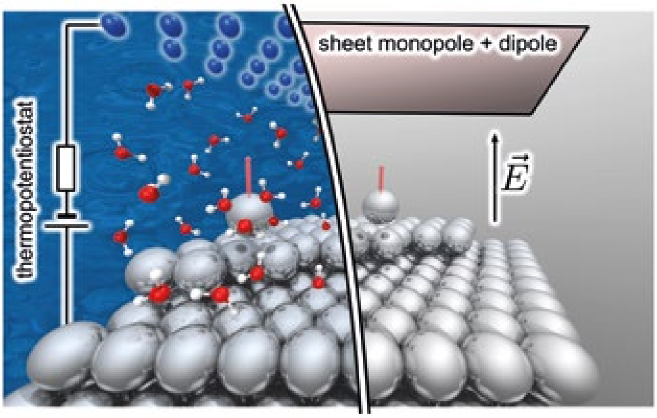

Fig. 1: *Schematic representation of dissolution (in electrochemical environment, left) and field evaporation (right) from a surface under the influence of an applied electric field.*

Exciting progress at the MPIE achieved over the last reporting period allows us now to realistically describe electric fields at charged surfaces from first principles. These approaches rely either on special electrostatic boundary conditions, as applied in, e.g., the *modern theory of polarization*, or model the electrified surface by a charged slab in a periodic cell and explicit compensating counter charges. Additionally, we introduced a "generalized dipole correction" that places an implicit computational counter electrode inside the vacuum region of the simulation cell, together with a discontinuity of the electrostatic potential [1]. The approach enables robust *ab initio* simulations of free surfaces in the presence of the extremely high (10^{11} V/m) electric fields that occur in atom probe tomography and similar experiments, and allowed us to explore and identify novel evaporation pathways [2].

When it comes to electrochemical experiments, the applied potential is held at a value that is constant on average at the macro-scale, but electronic and ionic charges fluctuate and transfer freely into and out of the region close to the interface, that is targeted by our simulations.

Therefore, when studying elementary processes at electrified interfaces by DFT we must treat the local surface charge not as constant, but as a thermodynamic degree of freedom with temperature-dependent fluctuations. Building on our finite electric field techniques, we recently introduced a "thermopotentiostat": a novel approach to control the electrode potential in *ab initio* molecular dynamics (AIMD) simulations [3]. By design, (i) it guarantees fundamental physical conservation laws such as the dissipation-fluctuation theorem, (ii) requires only quantities that are either easily accessible in DFT or are known from the specific computational setup, and (iii) it is straightforwardly implemented in any density-functional theory code. In fact, the thermopotentiostat has been implemented by us in VASP and in lammps.

Another technologically highly relevant field are semiconductor and (oxide) electrode materials as resulting e.g. from corrosion. Many of these materials show spontaneous polarization, which is not accounted for in conventional DFT approaches. Our recently developed generalized passivation method [4] ensures a correct description of the asymptotic bulk limit for pyroelectric materials, following a robust and quick convergence of total energies and opens the door towards an accurate description of the electronic surface band structure of such materials.

Our joint interdepartmental development activities benefit from mutual insights obtained in different areas of applications and scientific communities such as e.g. electrochemistry, surface science or semiconductor devices. In order to foster exchange also at an international level, for April 2020 we planned a three-day workshop at Ringberg castle on "*Electrified solid/water interfaces – theory meets experiment*", in order to bring together leading scientists from the areas of electrochemistry, solvation and spectroscopy. Due to the CoViD-19 situation, this workshop has been rescheduled to May 2022.

References

1. Freysoldt, C.; Mishra, A.; Ashton, M.; Neugebauer, J.: Phys. Rev. B 102 (2020) 045403.
2. Ashton, M. W.; Mishra, A.; Neugebauer, J.; Freysoldt, C.: Phys. Rev. Lett. 124 (2020) 176801.
3. Deißenbeck, F.; Freysoldt, C.; Todorova, M.; Neugebauer, J.; Wippermann, S.: Phys. Rev. Lett. 126 (2021) 136803.
4. Yoo, S.-H.; Todorova, M.; Wickramaratne, D.; Weston, L.; Van de Walle, C.G.; Neugebauer, J.: npj Comput. Mater. 7 (2021) 58.

Software engineering, development and digitalization

M. Hassani[1], M. Kühbach[2], N. Cautaerts[3], T. Hickel[1]

[1]CM, [2]MA, [3]SN

In 2020, an interdepartmental software task force (STF) was formed to serve as a forum for discussion on topics related to software development and digital workflows at the MPIE. A central goal was to facilitate interdepartmental collaboration by co-developing and integrating workflows, aligning internally developed software, and rolling out digital services. Trainings and workshops were organized in order to engage the entire MPIE community. Some highlights of the STF activities will be outlined in the following.

Each department presides over various independent software projects at various stages of development, the main ones being pyiron (CM) for atomistic simulations, DAMASK (MA) for modelling crystal plasticity, PARAPROBE (MA) for analysing atom probe tomography data, and TEMMETA (SN) for analysing transmission electron microscopy data. Since reproducibility is a central issue for all experimentalists and modellers, integration efforts centred on pyiron (see p. 72), which provides a platform for creating reproducible workflows. Mediated by interactions within the STF, the developers of DAMASK (see p. 73) and pyiron collaborated to create a common interface, thereby laying the foundations for direct coupling of atomistic simulations and continuum mechanics. Similarly, progress was made to integrate common TEMMETA workflows into pyiron, but for practical use cases graphical user interfaces must be developed, which is an ongoing effort.

The Jupyter notebooks employed here are becoming increasingly popular tools for creating such kind of advanced data analysis workflows. To reduce the barrier to experiment with these tools and to provide an environment of software packages for all researchers at the MPIE a jupyterhub server was launched in the CM department. The server allows MPIE members to perform digital workflows via a web browser. The deployment of the server utilizes in background a network of docker containers, which on the one hand ensures the reproducibility of the workflows via conda environments, and on the other hand takes care of the user authentication and http communications (Fig. 1).

In the SN department, an eLabFTW instance (an electronic lab notebook application) was launched to digitize the documentation of experimental workflows. With the help of QR codes that link to eLab entries, the history of samples and experiments performed on them is transparent and accessible to others after the creator of the sample leaves the institute. Through its python API, information on eLabFTW is accessible from jupyter notebooks and can be integrated with analyses. These workflows were presented outside the institute at multiple conferences and workshops.

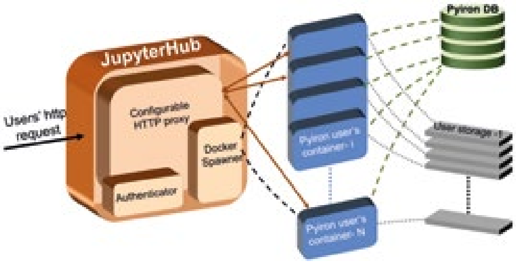

Fig. 1: *The structure of the docker network deployed in the cmserver02. Jupyter docker container authenticates users, and spawns Jupyter containers with preconfigured conda environment.*

Within the MPIE, STF organized three well received public events: (i) a seminar on electronic lab notebooks that featured an invited presentation by the developer of eLabFTW, (ii) a seminar demonstrating cmserver02 with a series of digital workflows featuring the simulation toolkits from the CM and MA departments, and (iii) a workshop on data visualization with python featuring a set of hands-on tutorials for plotting and image analysis (Fig. 2).

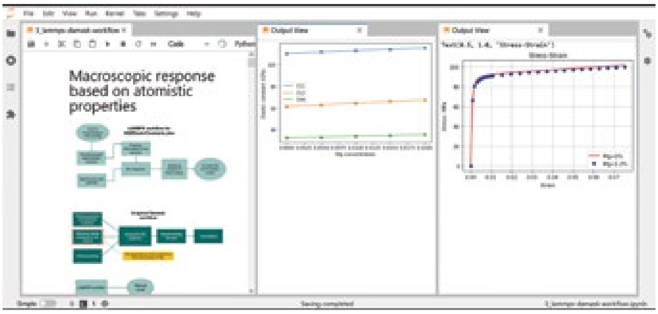

Fig. 2: *Snapshot of Jupyter notebook on the in-house jupyter hub server. The displayed DAMASK simulation determines the elas-to-plastic response of an Al-Mg alloy based on the properties obtained from a LAMMPS simulation.*

STF also organized internal meetings for knowledge exchange featuring talks on modern software development tools such as docker containerization, cython, E-Lab notebooks, Flask APIs, the numba and Dask packages, and CUDA programming in python. These meetings, as well as the workshops and hands-on-tutorials impressively demonstrated the practical potential of the developed hardware and the software infrastructure for interdepartmental collaborations.

Ab initio thermodynamics

T. Hickel, C. Freysoldt, L. Huber, F. Körmann, M. Todorova, J. Neugebauer

CM

The prediction of materials properties with *ab initio* based methods is a highly successful strategy in materials science. While the working horse density functional theory (DFT) was originally designed to describe the performance of materials in the ground state, the extension of these methods to finite temperatures has seen remarkable breakthroughs. This is needed, since many functional and structural mechanisms as well as the stability of relevant phases dramatically change with temperature.

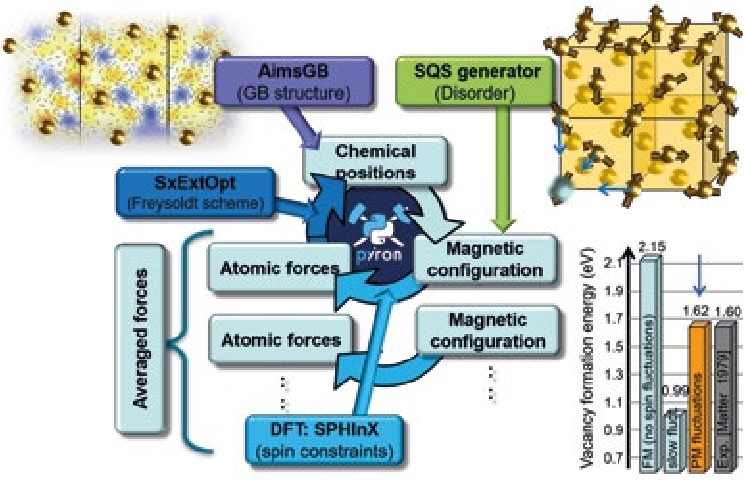

Fig. 1: *Simulation protocol for the atomic relaxation next to defects taking temperature dependent magnetic excitations into account.*

Since the foundation of the CM department in 2005, the MPIE has been a major driving force for these developments. It is systematically developing and exploring novel *ab initio* strategies to compute the vibrational, magnetic, electronic and configurational entropy contributions to Gibbs free energies, as well as their non-adiabatic coupling phenomena. These activities received a lot of attention in the scientific community and belong to the most-cited papers at the MPIE. Some major strategies in *ab initio* thermodynamics that have been substantially advanced in the last years are:

- Complex approaches to lattice vibrations well beyond the harmonic limit such as thermodynamic integration using Langevin dynamics (TU-TILD and TOR-TILD) have been systematically used for the determination of the melting temperature of metals. Based on high-throughput studies, a simulation protocol was developed that yields an uncertainty of the melting point below 5 K [1]. In order to achieve a high numerical efficiency, interatomic potentials had to be employed within these methods. The application of moment-tensor potentials (MTP) has made it recently possible, to describe the large configurational space of multicomponent alloys with high predictive power.

- Magnetic fluctuations at elevated temperatures and the resulting disorder above the ordering temperature are treated within the spin-space averaging (SSA) approach. Since this requires a large number of well-defined spin configurations, an accurate and efficient method for the spin-constrained DFT calculations was developed in the CM department [2]. The power of the approach was demonstrated for the atomic forces determining vacancy formations and diffusion in Fe-based alloys (Fig. 1).

These methodological breakthroughs became only possible due to the large advances in our in-house-developed platform pyiron (see p. 72), which provided the basis for rapid implementation and testing of the underlying complex simulation workflows. Thanks to these developments, it became possible to easily integrate distinctly different computer codes and handle huge numbers of separate calculations on heterogeneous computer architectures thus allowing rapid prototyping of complex and highly advanced simulation protocols. This provided also the basis to extend the *ab initio* thermodynamics in the CM department from the description of bulk phases to an analysis of the temperature and composition dependent stability of defects. Applications to stacking faults and grain boundaries have revealed unexpected trends and new interpretations of experiments. The investigations gave rise to the concept of defect phase diagrams, which is now the fundamental basis of the collaborative research center 1394 (see p. 76).

References

1. Zhu, L.F.; Janssen, J.; Ishibashi, S.; Körmann, F.; Grabowski, B.; Neugebauer, J.: Comp. Mat. Sci. 187 (2021) 110065.
2. Hegde, O.; Grabowski, M.; Zhang, X.; Waseda, O.; Hickel, T.; Freysoldt, C.; Neugebauer, J.: Phys. Rev. B 102 (2020) 144101.

Pyiron – an integrated platform for materials simulations and data management

J. Janssen, L. Huber, T. Hickel, J. Neugebauer

CM

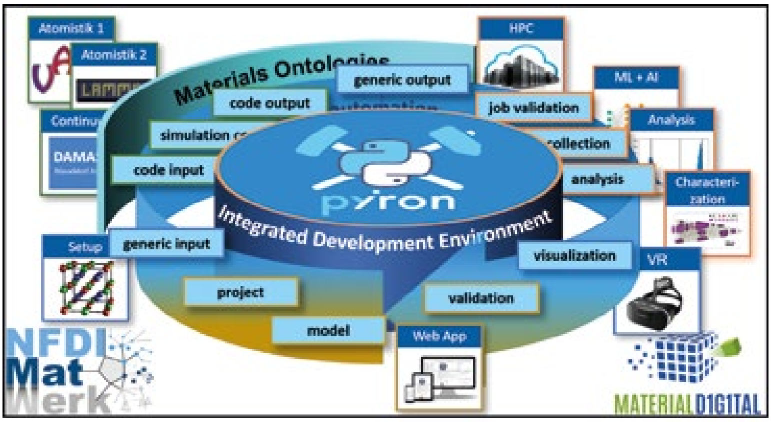

Fig. 1: *Generic workflow for complex simulation protocols in pyiron. Within the pyiron environment a large set of tools becomes accessible, while the technicalities of the interfaces are shielded from the user.*

The development of pyiron started in 2011 in the CM department to foster the implementation, rapid prototyping and application of the highly advanced fully *ab initio* simulation techniques developed by the department. The pyiron platform bundles the different steps occurring in a typical simulation life cycle in a single software platform and provides both developers and users an intuitive and easy to use interface that shields them from the underlying computationally efficient but highly complex data and job management concepts. Using from the beginning concepts such as generic rather than job specific input and output parameters allows incompatible computer codes such as Vasp, Sphinx, Lammps or Damask to directly communicate data thus making it easy for developers to build and test complex simulation protocols (Fig. 1). The advanced job management system allows an easy upscaling from interactive jupyter notebooks to high performance computing handling thousands of jobs.

In the last reporting period, a large number of new simulation tools and codes have been adapted to pyiron. To streamline the adaptation process of new tools and to make it also easy to our external collaborators and contributors, significant effort was made to render the underlying architectural concepts fully modular and easy to access by developers. These recent developments, together with the design criteria in terms of a generic input/output language, decentralized data management concepts and rapid upscaling from interactive development to high performance computing (HPC), which made pyiron so powerful in creating advanced materials science simulation workflows, turned out to be also of great interest to other fields. Together with the experience the CM department has gathered in getting such a software platform accepted and used by the actual users in their everyday workflows brought us in a position to initiate and successfully apply for large scale national initiatives broadly related to digitalization and materials science. For example, the CM department was one of the founding members and is now in the management board of a large Platform MaterialDigital (see p. 80) financed by the Ministry for Science and Education (BMBF). This expertise was also essential in getting substantial funding within the NFDI (national research data infrastructure) (see p. 81). Next to these large initiatives, pyiron turned also out to be essential for the success in more individual third-party grants.

The highly flexible architecture of pyiron and its strong focus on user friendliness and extendibility made it also attractive as data and workflow management tool for experimental activities in-house (see p. 70) and in large cooperation networks such as the CRC 1394 on Defect Phase Diagrams (see p. 76). Pyiron also turned out to be highly useful in designing and organizing interactive hands-on workshops on recent developments in materials simulations. Examples are workshops with more than 200 participants on linking atomistic calculations with CALPHAD concepts [1] or on machine learning potentials [2] where participants performed all steps from constructing representative density functional theory (DFT) data sets, via performing the fitting and testing of the potentials up to computing key materials quantities.

Thus, over the last reporting period pyiron has developed from a specialized and locally employed tool to a generic and widely used software platform that enabled the CM department to successfully initiate and participate in large scale networks as well as to initiate international collaborations and foster productivity.

References

1. https://pyiron.org/news/workshops/2020/11/10/workshop-on-software-tools-from-atomistics-to-phase-diagrams.html
2. https://pyiron.org/potentials-workshop-2021/README.html

The Düsseldorf Advanced Material Simulation Kit: DAMASK

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[3]School of Materials, University of Manchester, UK, [4]Chemical Engineering & Materials Science, Michigan State University, USA

Crystal plasticity modelling has gained considerable momentum in the past 20 years [1]. Developing this field from its original mean-field homogenization approach using viscoplastic constitutive hardening rules into an advanced multi-physics continuum field solution strategy requires a long-term initiative. The group “Theory and Simulation” of Franz Roters is working in this field since 2000. Code development during the last years was coordinated by the group “Integrated Computational Materials Engineering” headed by Martin Diehl (see p. 39).

DAMASK has developed from a plain crystal plasticity user subroutine into a modular multi-physics crystal plasticity simulation package. The capabilities of DAMASK have been described in an overview paper [2] together with numerous usage examples including contributions from many international users of DAMASK. The paper was published in 2019 and was by now cited almost 150 times, indicating the popularity of DAMASK in the computational materials science community.

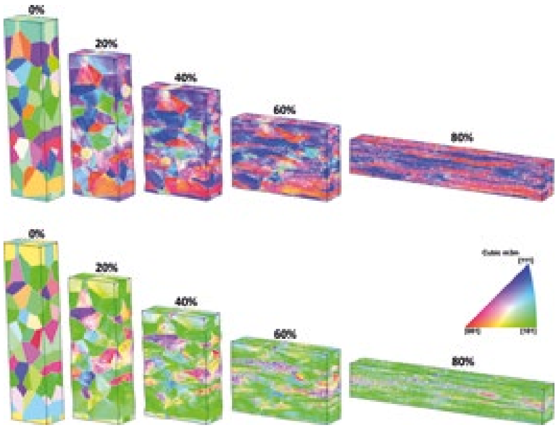

Fig. 1: *DAMASK simulation result for plane strain compression; top row: IPF parallel loading (vertical) direction, bottom row: IPF parallel stretching (horizontal) direction.*

The work after the publication of the overview paper focused on the development of DAMASK 3. This new version features three major improvements: 1. a modularization of the code; 2. new self-explanatory formats for input/output; 3. a python library for pre- and post-processing.

The main aim of the code refactoring was a consistent and decoupled implementation of the different physics (crystal plasticity, damage, temperature, etc.). The new structure of the code strictly separates the different field problems and their solvers but at the same time implements the functionality for interaction among the fields necessary for the fully coupled treatment of multi-field problems. As a side effect, we were able to decrease the memory footprint of DAMASK and increase the performance of the code, which is now fully parallelized.

The use of standardised formats for in- and output necessitated from the fact, that with the growing capabilities and speed of DAMASK, postprocessing became a bottleneck in the overall simulation process. For this reason, HDF5 [3] was chosen as the base for a flexible output format. HDF5 is a widely used structured binary format that also allows storing any kind of meta data together with the actual simulation results. All input files have been changed to the YAML [4] format. YAML is a human readable format used for structuring configuration data. In addition, both changes strengthen the use of DAMASK simulation data following the FAIR (Findable, Accessible, Interoperable, and Re-usable) principle.

The tools for pre- and post-processing originally provided in the form of shell scripts are now cast into a Python library. This change allows to store complete simulation workflows in the form of Jupiter notebooks for easy documentation and reuse. Usage examples as well as the full documentation of the library can be found on the newly released DAMASK website (https://damask.mpie.de).

DAMASK is developed as free and open source software and contributions in the form of additional features or bug fixes are welcome. The full sources are available at our GitLab repository (https://git.damask.mpie.de).

Ultra-high-resolution high deformation simulations enabled by the recent DAMASK improvements give unprecedented insights into the deformation behaviour of crystalline materials.

References

1. Roters, F.; Eisenlohr, P.; Hantcherli, L.; Tjahjanto, D.D.; Bieler, T.R.; Raabe, D.: Acta Mater. 58 (2010) 1152.
2. Roters, F.; Diehl, M.; Shanthraj, P.; Eisenlohr, P.; Reuber, C.; Wong, S.L.; Maiti, T.; Ebrahimi, A.; Hochrainer, T.; Fabritius, H.-O.; Nikolov, S.; Friák, M.; Fujitai, N.; Grilli, N.; Janssens, K.G.F.; Jia, N.; Kok, P.J.J.; Ma, D.; Meier, F.; Werner, E.; Stricker, M.; Weygand, D.; Raabe, D.: Comput. Mater. Sci. 158 (2019) 420.
3. The HDF Group. Hierarchical Data Format, version 5, 1997-2021. https://www.hdfgroup.org/solutions/hdf5/
4. YAML.org Website, https://yaml.org/

International Max Planck Research School for Interface Controlled Materials for Energy Conversion IMPRS-SurMat

E. Gattermann[1], S. Wippermann[2]

[1]RCO, [2]GO

The IMPRS-SurMat is a structured 3-years doctoral program existing since 2004. Aims of the school are:

- Recruiting and training of international young scientists
- Formation and intensification of collaboration between the participating institutes
- Strengthening of interdisciplinarity across the faculties

In the report period, the school is in the second half of its third running period and hence well-established. The running periods of 6 years end with an external evaluation that the IMPRS-SurMat successfully passed in 2009 and 2014.

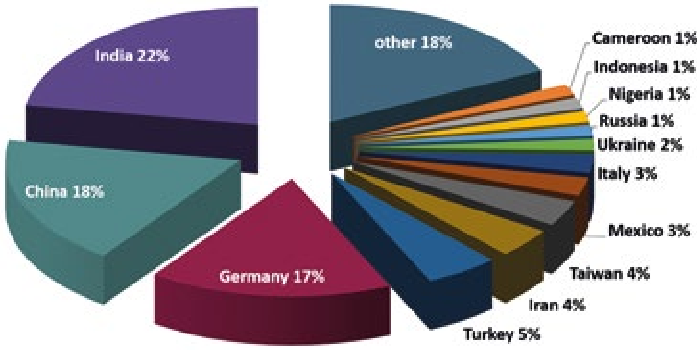

Fig. 1: *Home countries of doctoral students.*

Partner institutes of the school are the MPI für Chemische Energiekonversion and the MPI für Kohlenforschung, both Mülheim/Ruhr, the Ruhr-Universität Bochum and the Universität Duisburg-Essen. There are 26 PIs and about 50 students in the school.

The scientific subject “Interface Controlled Materials for Energy Conversion” combines the individual strengths of these partners and focuses on five connected topics:

- Microstructure and physics of defects
- Hydrogen uptake in materials
- Oxygen reduction and evolution reactions
- Materials for future energy systems
- Degradation mechanisms and life extension of materials

Main funding of the IMPRS-SurMat comes from the Max Planck Society, but all partner institutes make relevant contributions and additional financial support could be gained by the state North Rhine-Westphalia.

The school is open for students from all over the world with a master’s degree in materials science, physics, chemistry or related subjects and offers funding for 3 years (working contracts or scholarships). In the years 2019-2021, 28 new students were accepted into the program. Thereby, 197 doctoral students entered the school in total. They originated from 28 different countries, to a significant degree from Asia, but also from all other regions of the world. 32 % of the doctoral students are female scientists.

The Corona pandemic was without any doubt the biggest challenge the school had to face. All curricular events as well as the recruitment of new doctoral students were initially disrupted. Therefore, in the year 2020 the school’s program was successfully adapted into well-running online formats.

The main part of the curriculum are the lectures that take part in the first two years of the doctoral project work. The students attend 5 lectures on the following topics:

- Material characterization
- Microstructure and mechanical properties
- Interface chemistry
- Electrochemistry and corrosion
- Simulation and modelling

The annual retreat at an external venue had to be cancelled on short notice in spring 2020, but could be held online in 2021. In this format, all PhD students present their projects and current results. Every two years, the supervisors are also invited to attend.

Further components of the curriculum are the Welcome Days for new doctoral students, first introduced in 2019, soft skills trainings such as project management, presentation techniques and scientific writing, the possibility to attend German courses, and support for further career planning such as leadership training and alumni talks.

The IMPRS-SurMat accompanies the students on their way to the doctoral degree and ensures that they are provided with a close and fruitful supervision. Once per year, the doctoral students send a short report about the progress of their work to the program coordinator. Subsequently, Thesis Advisory Committee (TAC) meetings take place to discuss the results and plan the next working steps.

Since 2004, 121 students have successfully finished their doctorate within the IMPRS-SurMat, 24 of them with distinction. The doctoral work requires on average between 3 and 4 years with a genuinely high scientific output. 527 journal articles were published, as well as more than 350 poster and 380 oral presentations at national and international conferences and workshops.

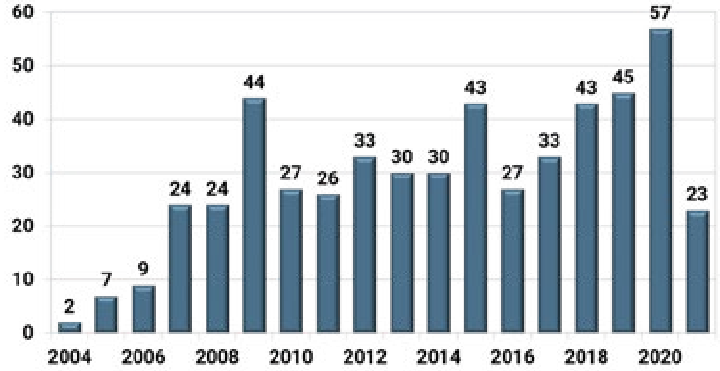

Fig. 2: *Articles in peer reviewed journals.*

The new alumni network started in 2020. Joining the alumni network is easily done by registering online or sending the completed registration form via email or post. The alumni are encouraged to stay in touch via a mailing list or the LinkedIn profile of the IMPRS-SurMat. The alumni network provides the following activities:

Fig. 3: *Retreat at Ringberg Castle in 2019.*

- A newsletter is sent via the mailing list to all registered alumni once per year.
- On request, the program coordinator establishes contact between doctoral students and alumni for job shadowing and career mentoring.
- The alumni talk series “Career Paths after PhD presented by Alumni” takes place several times per year.
- An alumni symposium with scientific and career talks is planned for the time after the corona pandemic.
- Thus, the alumni network is a relevant part of the career development activities of the IMPRS-SurMat.

After its total designated runtime of 18 years, the IMPRS-SurMat will conclude at the end of the current running period in December 2021. Due to this termination, the management of the MPIE decided to keep the well-proven format of an IMPRS but to restart with a new IMPRS. In June 2021, the MPIE together with the partner institutes of IMPRS-SurMat applied for the new International Max Planck Research School for Sustainable Metallurgy – from Fundamentals to Engineering Materials, the IMPRS-SusMet. The Max Planck Society fully approved the proposal in November 2021. The start of the IMPRS-SusMet is planned for January 2022.

Structural and chemical atomic complexity – from defect phase diagrams to material properties

J. Neugebauer[1], E. Bitzek[1], G. Dehm[2], T. Hickel[1], L. Huber[1], D. Raabe[3], F. Roters[3], C. Scheu[4], M. Todorova[1]

[1]CM, [2]SN, [3]MA, [4]NG

Teams from RWTH Aachen University, MPIE and FAU Erlangen-Nürnberg set up a new Collaborative Research Center (CRC) on establishing a framework to construct defect phase diagrams and to employ these diagrams to design materials with tailored properties (Fig. 1). The CRC1394 is funded for the first 4 years by the German Research Foundation (DFG) with approx. 2 Mio/year supporting 18 scientific projects with 20 postdocs and PhD students. It is headed by S. Korte-Kerzel (RWTH Aachen University, spokesperson) and J. Neugebauer (MPIE, deputy spokesperson). Three MPIE departments and the independent research group NG are actively involved and contribute to 8 projects.

The CRC aims to establish a new conceptual and methodological framework that combines two fundamental concepts in materials design (Fig. 2): Thermodynamic phase diagrams that describe the conditions at which specific phases become stable, with the structure and stability of lattice defects that determine key material properties such as mechanical strength, protection against failure mechanisms or corrosion resistance. The new holistic concept, therefore, extends the concept of bulk phase diagrams to defect phase diagrams, allowing materials physicists and engineers to identify suitable conditions where defect states with targeted properties can be realized.

A key prerequisite to constructing such defect phase diagrams is the availability of advanced experimental and theoretical tools to identify and predict the often highly complex chemical and atomistic structures that characterize the various defect states. Essential to achieve this are highly sophisticated experimental tools, such as high-resolution transmission electron microscopy (HR-TEM) or atom probe tomography (APT), but also correlative approaches combining several techniques, as developed over the last years at the MPIE in the MA and SN departments, to provide both atomistic and chemical resolution at the same sample position. The advanced *ab initio* based approaches developed by the CM department allow to include all relevant entropic contributions and thus to provide accurate Gibbs energies for defects at finite temperatures. Large-scale atomistic simulations and crystal plasticity modelling in the CM and MA departments allow to assess the impact of defect phases at the microstructure level. Together with the extensive expertise at RWTH Aachen University in, e.g., synthesizing high-quality samples and measuring the impact of defects on various mechanical and corrosion properties, the consortium of researchers will also be able to construct mechanism maps to identify the impact of specific defect states on macroscopic mechanical or corrosion properties.

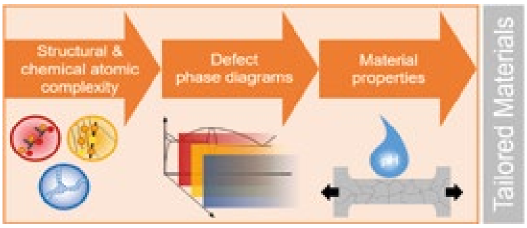

Fig. 1: *Vision of the CRC: Description of the structural and chemical complexity of defects at the atomic scale in defect phase diagrams together with their mechanical and corrosion properties will provide a Materials Design Toolbox enabling materials scientists and engineers to design tailored materials.*

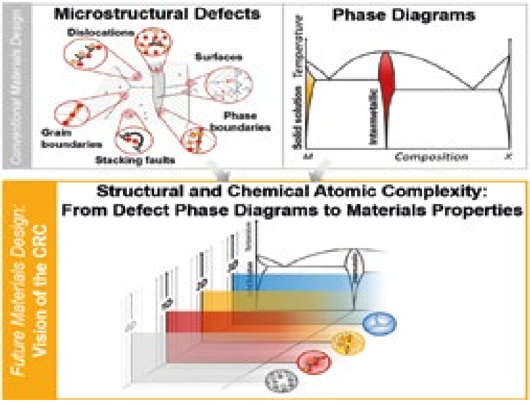

Fig. 2: *Extension of materials design from the currently decoupled approaches considering microstructural defects and phase diagrams towards a comprehensive framework considering both aspects in terms of the full structural and chemical complexity of defects at the atomic scale and their thermodynamic stability.*

This structural and chemical complexity at the atomic scale naturally exists in all materials. The CRC will provide new quantitative descriptors of the local structure and chemistry of defects, which govern materials' bulk properties. Their availability will allow a paradigm change in the physical description of metallic materials and will provide a powerful toolbox for future design of engineering materials with tailored properties regarding both, mechanical and corrosion performance.

References

1. Korte-Kerzel, S.; Hickel, T.; Huber, L.; Raabe, D.; Sandlöbes-Haut, S.; Todorova, M.; Neugebauer, J.: Int. Mater. Rev. 66 (2021).

Compositionally complex alloys – high entropy alloys

F. Körmann[1], Y. Gong[1], J. Neugebauer[1], Z. Li[2], Y. Ma[2], D. Raabe[2],
N.J. Peter[3], S. Wolff-Goodrich[3], C. Liebscher[3], G. Dehm[3]

[1]CM, [2]MA, [3]SN

Multicomponent alloys such as compositionally complex alloys (CCA) or high entropy alloys (HEA) have opened various avenues for exploring new materials beyond the limits of traditional alloys based on one or two main components. In 2017, the German Research Foundation (DFG) has established the six-years running Special Priority Programme (SPP) 2006 "Compositionally Complex Alloys – High Entropy Alloys" to support the research on HEAs and CCAs. Several projects in Germany are supported by this large-scale DFG program, and the MPIE is with several granted projects very actively and successfully participating:

(1) "Design and mechanical properties of compositionally complex alloys from twinning-induced towards bidirectional transformation-induced plasticity" (Y. Gong, F. Körmann, Y. Ma, Z. Li, D. Raabe). In the first project phase, the transformation induced plasticity (TRIP) effect has been successfully introduced into interstitially alloyed face centered cubic (FCC) CrCoNiMnFe-based HEAs utilizing a combined *ab initio*-experimental approach. Three quinary HEAs have been selected from *ab initio* calculations, experimentally prepared and investigated [1]. The atomistic calculations highlight the high efficiency of the developed coupled *ab initio*-experimental approach developed within the first project phase for developing mechanically high-performing, low-stacking fault energy (SFE), interstitially alloyed CCAs. The follow-up project builds on the successful first project phase and aims at further significantly enhancing the strength-ductility combination of quinary CCAs by introducing the bidirectional TRIP effect into interstitially alloyed CCAs.

(2) In "Tailored precipitation strengthened, compositionally complex FeAlCr (Mn, Co, Ni, Ti) alloys for high temperature applications" (C. Liebscher, K. Pradeep / IIT Madras, J. Schneider / RWTH Aachen University) we developed new combinatorial synthesis techniques based on thin film libraries to efficiently screen the unexplored composition space Al-Cr-Fe-Ni-Ti. We identified a global trend that alloys with Al contents of ≥20 at.% are primarily comprised of a disordered bcc (A2) matrix strengthened by ordered B2/L2$_1$-type precipitates. Based on this rapid screening, low density (~6.4 gcm^{-3}) alloys with a nanostructured duplex dual-phase microstructure were developed showing tensile yield strength of up to ~220 MPa at 900°C (see fig. 1). The most promising alloy with composition $Al_{20}Cr_{20}Fe_{35}Ni_{20}Ti_5$ (at.%) even exhibits excellent tensile creep strength at 800°C resisting similar stress levels as comparable ferritic superalloys tested at 700°C [2]. Deep microstructural characterization revealed that the discrete precipitate morphologies and volume fractions, as well as the supersaturated solid solution are responsible for equipping these alloys with outstanding high temperature properties.

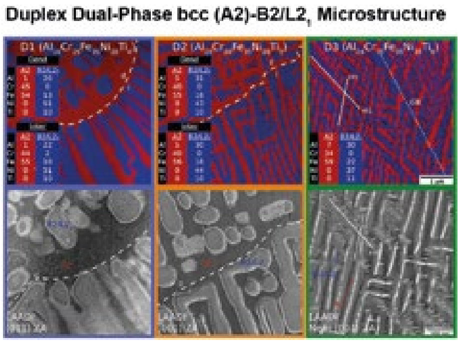

Fig. 1: *Scanning transmission electron microscopy (STEM) investigation of duplex dual-phase microstructures for alloys with 16, 20 and 24 at.% (from left to right).*

(3) In the first funding period (N.J. Peter, E.A. Jägle, G. Dehm) we established a synthesis route for particle-reinforced compositionally complex alloys (p-CCA) by powder atomization and additive manufacturing to reveal their microstructure evolution and understand their mechanical properties. We chose to work in the alloy system CoCrFeNi(-Al,-Mn). Powder of all alloys could be produced successfully and were optimized in terms of flowability by controlled addition of SiO2 nanoparticles [3]. While the quaternary base alloy consisted of a single-phase (A1) microstructure, Al additions always resulted in a A2/B2 decomposition pattern [4]. Through microcompression experiments we were able to quantify the effect of different microstructural features, such as intentionally introduced oxide precipitates, on the incipient plasticity of p-CCA alloys using cast reference material. For the second funding phase of the SPP, we build on the established processing routes, alloy design and understanding of deformation mechanisms to study in greater depth the performance of p-CCA at cryogenic temperatures and under hydrogen loading conditions.

References

1. Wu, X.; Li, Z.; Rao, Z.; Ikeda, Y.; Dutta, B.; Körmann, F.; Neugebauer, J.; Raabe, D.: Phys. Rev. Mat. 4 (2020) 033601.
2. Wolff-Goodrich, S.; Haas, S.; Glatzel, U.; Liebscher, C.H.: Acta Mater. 216 (2021) 117113.
3. Gärtner, E.; Jung, H.Y.; Peter, N.J.; Dehm, G.; Jägle, E.A.; Uhlenwinkel, V.; Mädler, L.: Powder Technol. 379 (2021) 585.
4. Jung, H.Y.; Peter, N.J.; Gärtner, E.; Uhlenwinkel, V.; Dehm, G.; Raabe, D.; Jägle, E.A.: Addit. Manuf. 35 (2020) 101337.

Analysis of the stability of high entropy alloys by dewetting of thin films

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The project AHEAD funded by the German and French Research Foundations (DFG-ANR) centered on phase stability of high entropy alloy (HEA) thin films by systematically manipulating 1D and 2D defects. The joint French-German team with its expertise in different fields of materials science (combinatorial thin film synthesis, microstructure physics, thermodynamics and mechanics of materials) worked from 2017 – 2020 on the following fundamental issues of HEA films: (i) HEA phase stability, (ii) phase evolution and corresponding kinetics including influence of composition, defects (dislocations, interfaces, grain boundaries) and dimensional constraints on phase stability, (iii) grain growth and texture, (iv) dewetting kinetics and morphologies, (v) temperature and microstructure-stress evolution, (vi) plastic deformation, (vii) thermo-mechanical fatigue mechanisms and lifetimes. The material system was mainly centered around fcc-(Mn)CrFeCoNi.

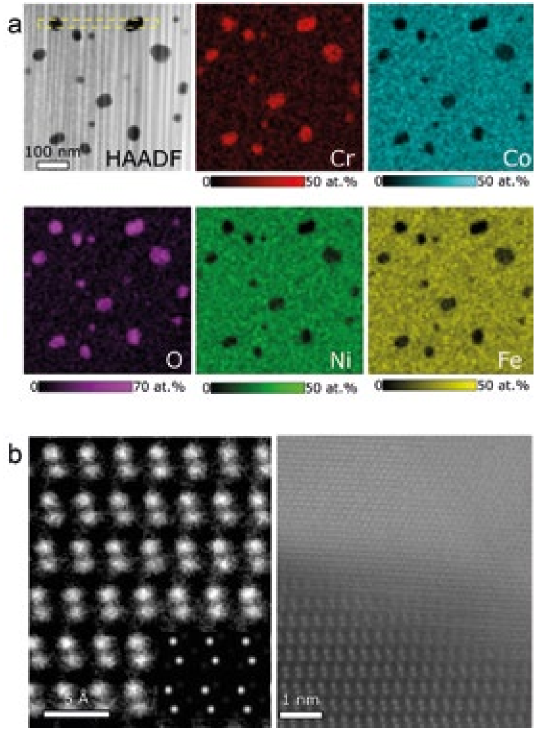

Fig. 1: *(a) Scanning transmission electron microscopy (STEM) Z-contrast image (high angle annular detector: HAADF) showing the distribution of Cr_2O_3 nanoparticles and corresponding elemental maps by STEM-energy dispersive X-ray spectroscopy. (b) Atomic resolved STEM-HAADF and annular bright field images of the oxide particles identified as Cr_2O_3 with the R-3c space group [3].*

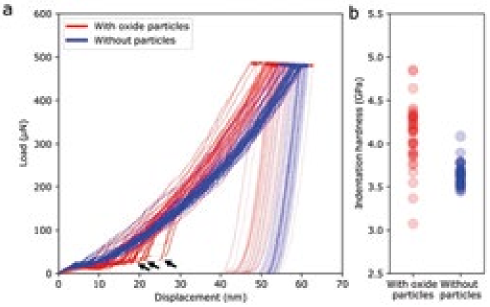

Fig. 2: *(a) Nanoindentation data and b) distribution of nanohardness values show a higher film hardness for the quaternary films with Cr_2O_3 particles (red curves and data points) [3].*

In this short overview we focus on thin film microstructure, oxidation and mechanical properties of CrFeCoNi. Room temperature deposited, iso-quaternary films consist of two-phases, the fcc matrix and a small amount of sigma phase. The nanocrystalline matrix (grain size 30-100 nm) has a <111> fiber texture. The sigma phase dissolves upon annealing between 700 and 1150 °C [1].

Chromium oxide forms at the surface of the film even in the most reducing conditions of annealing. It is the thinnest when annealing takes place under ultra high vacuum (UHV) or in pure hydrogen. We have further dealt with this layer to obtain reliable measurements of the entire film microstructure and stability under dewetting [2]. We have also taken advantage of this oxide formation [3] to study the role of oxide precipitates within the matrix on the mechanical properties of the film (Fig. 1). Hardness measurements of pristine and Cr-oxide dispersion strengthened CoCrFeNi alloy thin films revealed a 13 % increase in hardness (Fig. 2) due to 1.5 vol% of 12 nm large oxide particles revealing that the lack in high temperature strength of this alloy can be improved by disperoids [3].

In addition, we analyzed the dewetting behaviour, which starts with a competition between grain growth and grain boundary grooving and is surprisingly similar as for fcc elemental films [2].

References

1. Kini, M.K.; Lee, S.; Savan, A.; Breitbach, B.; Addab, Y.; Lu, W.; Ghidelli, M.; Ludwig, A.; Bozzolo, N.; Scheu, C.; Chatain, D.; Dehm, G.: Surf. Coat. Technol. 410 (2021) 126945.
2. Addab, Y.; Kini, M.K.; Courtois, B.; Savan, A.; Ludwig, A.; Bozzolo, N.; Scheu, C.; Dehm, G.; Chatain, D.: Acta Mater. 200 (2020) 908.
3. Lee, S.; Chatain, D.; Liebscher, C.H.; Dehm, G.: Scr. Mater. 203 (2021) 114044.

Interdisciplinary Centre for Advanced Materials Simulation

T. Hickel, J. Neugebauer

CM

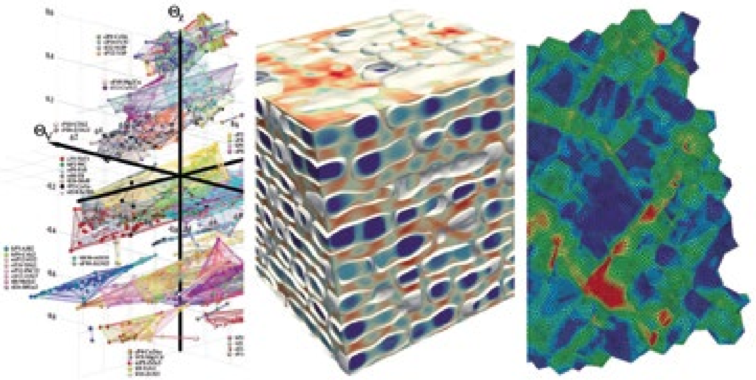

Fig. 1*: The three departments of ICAMS combine simulations at different scales, resulting into (from left) atomic structure maps, phase field and finite element simulations as typical images.*

The Interdisciplinary Centre for Advanced Materials Simulation (ICAMS), which has been initiated in 2005 by the MPIE and has been founded in 2008, has become one of the leading institutions for scale bridging materials modelling. It includes the departments for "Atomistic Modelling and Simulation" (headed by R. Drautz), "Scale Bridging Thermodynamic and Kinetic Simulation" (I. Steinbach), and "Micromechanical and Macroscopic Modelling" (A. Hartmaier) (Fig. 1). After the initial startup period of five years with substantial financial support from an industrial consortium, the state of North Rhine-Westphalia and the European Union, the institute has now become a regular unit within the Ruhr University Bochum with international visibility. It serves as an integral part of the Material Research Department within the university, but also of the Materials Chain of the University Alliance Ruhr. The latter combines the research activities in the field of materials science and technology at the three Ruhr Area universities Ruhr-Universität Bochum, TU Dortmund University, and the University of Duisburg-Essen.

Structurally, the MPIE is embedded into ICAMS via the Advanced Study Group (ASG) "*Ab initio* based modelling", which supports the multiscale concept in particular at the most fundamental scale, which is dominated by electronic interactions and individual atomic processes. The link between ICAMS and MPIE is further deepened within several large-scale projects funded by the German Research Foundation (DFG) that have been attracted in the past years: Within the Collaborative Research Centre (SFB/Transregio 103) "From Atoms to Turbine" joint research on the characterization of superalloys is performed. The MPIE was strongly participating in the Priority Program (SPP1713) "Chemomechanics" that was coordinated by I. Steinbach. Since 2018 the Research Unit ""Automated analysis and validation of interatomic potentials for application in Materials Science" is a joint effort to develop, implement and provide a transparent and comprehensive validation framework for models of the interatomic interaction. Various other projects, e.g., within the Max-Planck Research School SURMAT (see p. 74), have led to an increasing amount of PhD theses that are jointly supervised by members of both institutions. Also, lectures within the ICAMS Master of Science program "Materials Science and Simulation" (MSS) are partially held by MPIE members.

The highly productive scientific collaboration between researchers at ICAMS and MPIE is documented by a large number of publications, a significant amount of attracted third-party funding, and in particular by the joint scientific outreach. Several workshops (e.g. ADIS workshop 2020, Potentials workshop 2021, MCIC 2021 conference) and symposia at international conferences have been jointly organized. Regular retreats are performed together with the CM department. The ICAMS Advanced Discussions (Fig. 2) are meanwhile an established institution. ICAMS has recently extended its activities by establishing the new research group on "Scale-Bridging Simulation of Functional Composites" (A. Grünebohm) and "Materials Informatics and Data Science" (M. Stricker). This opens additional channels of interaction with all departments at the MPIE, which have already started and will further strengthen the fruitful exchange with ICAMS.

Fig. 2: *The audience during the ICAMS Advanced Discussions 2019 in Bochum.*

The innovation platform MaterialDigital

T. Hickel[1], F. Roters[2], J. Neugebauer[1]

[1]CM, [2]MA

The digitalization of materials science and engineering is currently addressed with highest priority in Germany. To ensure international competitiveness, there is a high motivation in German industry to implement novel and sustainable concepts to manage and share materials related data hosted on decentralized and highly diverse servers. The CM department has gained with the development of the pyiron environment (see p. 72) early an expertise in the design and dissemination of decentralized and materials oriented software solutions and tools. To bring in this expertise, the MPIE became one of the initiators and founding members of the innovation platform MaterialDigital (PMD).

Fig. 2: *One of the main goals of PMD is to establish a workflow infrastructure that provides a user-friendly interface to a large variety of tools.*

The PMD has started in July 2019 as a collaborative effort of five institutions representing the societies Fraunhofer, Helmholtz, Leibniz, and Max Planck as well as the Federal Institute for Materials Research and Testing (BAM). The Max Planck share is taken over by the CM department and headed by J. Neugebauer and T. Hickel. The initial phase of PMD is funded by the Federal Ministry of Education and Research (BMBF) with 2 million euros annually in order to support interested parties from the industrial and academic sectors in the implementation of digitization tasks for materials. Its mission is the establishment of a virtual materials data space and the systematization of the handling of hierarchical, process-dependent materials data.

Fig. 1: *The decentralized data storage concept of the platform material digital, which supports the sharing of workflow solutions instead of huge amounts of raw data.*

To this end, three major concepts are established by the PMD: (i) The storage and access to data will be performed in a decentralized infrastructure (Fig. 1), keeping data at the place where they have been generated. (ii) An ontological structure will be used as the basis of the information ecosystem to make data findable and accessible. (iii) Any processing of data will be performed in a generalized workflow environment to ensure the reproducibility of process chains that transform data to a particular set of outputs. In order for these concepts to be adopted by the community, the PMD also serves as an interaction and communication platform.

Using the large expertise gained with pyiron, the MPIE strongly contributes to these concepts and in particular to the workflow infrastructure (Fig. 2). The goal is to use the pyiron philosophy and architecture to provide scientists and engineers a user-friendly interface to a large variety of tools as well as access to standardized computational procedures that are based on complex connections between these tools. Since the CM department is also PI for the National Data Infrastructure (see p. 81), which is devoted to digitalization in academia, our mission is to ensure a consistency of workflow standards in the industrial and academic community.

In a second call, the BMBF has selected 13 projects from much more than 100 proposals to develop and implement strategies for the digitalization of dedicated user stories in materials science. Being strongly connect to PMD, they represent certain materials communities. The project StahlDigital has been selected to handle ontology-based interoperable workflows for the development and optimization of steel materials from the sheet metal production up to the crash safety of components. It is coordinated by F. Roters and contains among other partners contributions of the MA and CM departments.

A national research data infrastructure for materials science & engineering

T. Hickel, E. Bitzek, J. Neugebauer

CM

In 2016 the Gemeinsame Wissenschaftskonferenz (GWK) has decided to create a sustainable national infrastructure for research data. Throughout the whole scientific landscape of Germany up to 30 consortia will be funded with a total of up to 85 Mill. €/a for an initial period of five years. Headed by the Fraunhofer IWM institute in Freiburg, the proposal for the community Materials Science & Engineering (MatWerk, engl.: MSE) had been submitted in 2020 by 10 co-applicants, including the MPIE. Having received a grant within this framework can be considered a big success and demonstrates the grown importance of a research field that is central for the MPIE.

The initiative NFDI-MatWerk is a community-driven effort to structure materials data according to the FAIR principles, i.e., making them accessible, findable, interoperable and reusable. As daily research at the MPIE demonstrates, the inherent multiscale character related to strongly heterogeneous microstructures is a particular challenge for MSE data. In addition, the history of data evolution is important, since any process applied to a sample may change this microstructure and, thereby, its mechanical and functional performance. NFDI-MatWerk aims at tracking these various complex dependencies of materials data, while reducing the technological barriers to exploit them (Fig. 1).

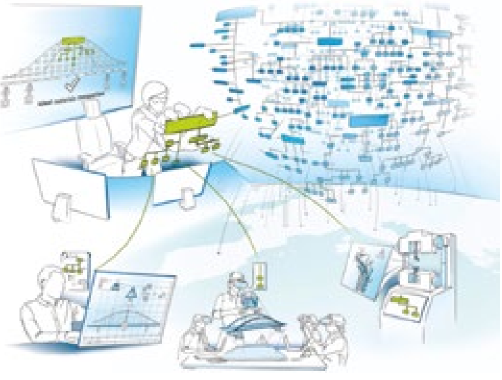

Fig. 1: *NFDI-MatWerk wants to make decentralized expertise integrable through a uniform data language and standardized interfaces.*

The most important objectives of NFDI-MatWerk are represented by Task Areas (TAs) and two of them are represented by group heads at the MPIE. E. Bitzek (see p. 37) is a co-speaker of the TA “Materials Data Infrastructure”, which will provide a reliable environment for the digital representation of materials data and metadata as well as services to easily store, share, search, and analyze them. T. Hickel is a co-speaker of the TA “Workflows and Software Development” that will use the in-house developed software platform pyiron (see p. 72) to establish a framework for sharing workflows in processing environments, for implementing automated experimental and modelling protocols with widespread tools and, therewith, for uniting workflows with the underlying materials data.

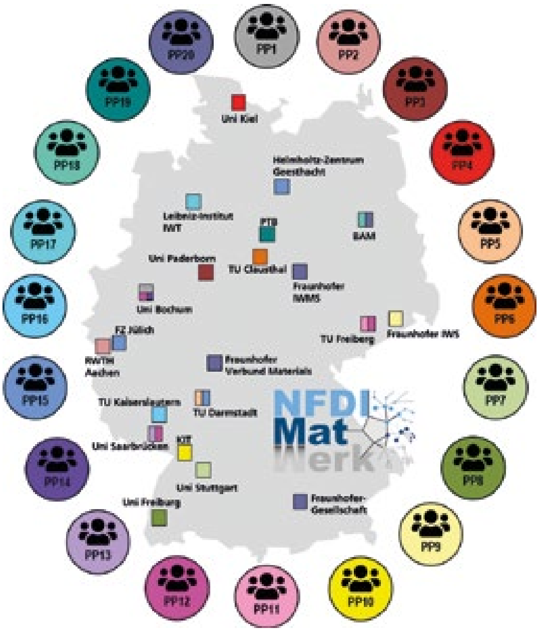

Fig. 2: *The geographic distribution of the participant projects (PP) of NFDI-MatWerk, which second the ten (co-) applicant institutions.*

A third TA will provide and integrate a unified materials ontology, that is represented through a graph database infrastructure. This enables data sharing as well as highly performant, complex search queries and analysis runs over distributed and decentral data sources (Fig. 1). The concepts developed in NFDI-MatWerk are permanently challenged by Infrastructure Use Cases defined and tested by a large number of participant projects (Fig. 2). Since employees at the MPIE are associated to several of these projects, it is ensured that the perspective of individual scientists at the MPIE (as well as many other institutes) will guide the developments within NFDI-MatWerk. In this way, the investment into NFDI-MatWerk is expected to pay off multiple times in terms of an infrastructure that supports the research at the institute.

More details: https://nfdi-matwerk.de

BiGmax network on big-data driven materials science

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J. Neugebauer[1], D. Raabe[3]*

[1]CM, [2]MA, [3]SN

The Max-Planck network on big-data driven material science, BiGmax, was established in 2017 to bundle efforts among 10 Max-Planck institutes and the Humboldt University in Berlin. The main research topics range from machine-learned mechanical and thermodynamic properties over data diagnostics in imaging and discovery of patterns, correlation and causality to promoting data accessibility and interpretability for algorithms. The MPIE has played an essential part in initiating the network, and, in part thanks to additional support drawn from the network, is now the largest single node, with activities from the MA, CM, and SN departments. In line with the general topics, our research focuses on pattern discovery and data standardization.

Data-rich experiments such as atom probe tomography (APT) or scanning transmission electron microscopy (STEM) provide large amounts of multi-dimensional raw data that encodes, via correlations or hierarchical patterns, much of the underlying materials physics. With modern instrumentation, data generation tends to be faster than human analysis, and the full information content is rarely extracted. We therefore work on automatizing these processes as well as on applying data-centric methods to unravel hidden patterns.

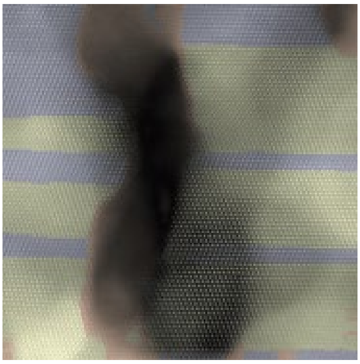

Fig. 1: *Automatically segmented HAADF-STEM image of Pd nanoparticles, highlighting twin domains (color-coded yellow, blue; red areas are featureless/amorphous).*

For instance, deep neural networks can be trained to disentangle mass spectra in APT, and thereby yield reproducible mappings to the chemical identity [1]. Likewise, crystallography-induced fluctuations in the intensity map on the detector can be assigned to crystallographic poles and hence reveal crystal orientation via machine-learning, trained on hundreds of synthetic data sets. This can even be expanded to track grain boundaries even when the underlying lattice is not resolved in the APT reconstruction [2]. Ordered binary phases, on the other hand, can be identified in pair-correlation functions by convolutional neural networks [3].

High-resolution STEM, on the other hand, may image crystallographic patterns directly. Within BiGmax, we developed an automatic segmentation based on local symmetry descriptors [4]. Fig. 1 shows the result of such a segmentation for Pd nanoparticles, where the twinning domain structure becomes visible at first glance. But even when the atomic lattice is not resolved, fitting to a neural network is a promising approach to coarse-grain *in situ* image series to a smooth phase-field representation, removing much of the shot noise along the way. Such a smooth, differentiable representation of the experimental data subsequently enables us to quantify processes at the coarse level, e.g. phase transformation kinetics.

Secondly, we work on standardizing data set descriptions and data formats to allow for an easier exchange of both data and analysis codes. While this looks like a technical detail at first sight, it is an essential prerequisite to bring advanced analysis tools to the finger-tips of the experimental scientist on a daily basis, similar to what has been achieved in modelling with e.g. the NOMAD/FAIRMAT project run by other partners within BiGmax [5]. For APT, moving away from commercial data formats to open-source, exchangeable binary formats based on HDF5 dramatically improves the processing speed and allows to build up complex, automatized workflows [6]. Similar progress is made for analysing complex 4D-STEM datasets, where in each scan point a full electron diffraction pattern is obtained. We developed an open-source workflow to determine the grain and phase orientation in nanomaterials, which is typically only possible with commercial software [7, 8]. To ensure adoption in the community and interoperability with other tools, we integrate our solutions into established open source analysis libraries.

References

1. Wei, Y.; Varanasi, R. S.; Schwarz, T.; Gomell, L.; Zhao, H.; Larson, D. J.; Sun, B.; Liu, G.; Chen, H.; Raabe, D.; Gault, B.: Patterns 2 (2021) 100192.
2. Wei, Y.; Peng, Z.; Kühbach, M.; Breen, A. J.; Legros, M.; Larranaga, M.; Mompiou, F.; Gault, B.: PLoS One 19 (2019) 1.
3. Li, Y.; Zhou, X.; Colnaghi, T.; Wei, Y.; Marek, A.; Li, H.; Bauer, S.; Rampp, M.; Stephenson, L.: npj Comp. Mater. 7 (2021) 8.
4. Wang, N.; Freysoldt, C.; Zhang, S.; Liebscher, C. H.; Neugebauer, J.: Microsc. Micronanal. (2021), in press.
5. https://nomad-coe.eu/home-coe
6. Kühbach, M.; Bajaj, P.; Zhao, H.; Çelik, M. H.; Jägle, E. A.; Gault, B.: npj Comp. Mat. 7 (2021) 21.
7. Cautaerts, N.; Rauch, E. F.; Jeong, J.; Dehm, G.; Liebscher, C. H.: Scr. Mater. 201 (2021) 113930.
8. Cautaerts, N.; Crout, P.; Ånes, H.W.; Prestat, E.; Jeong, J.; Dehm, G.; Liebscher, C.H.; arXiv:2111.07347

Leibniz-Max-Planck cooperation: carbon-metal oxide nanohybrids

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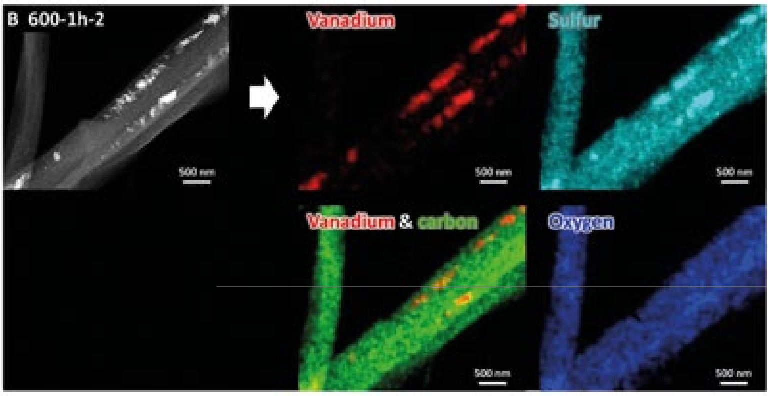

Fig. 1: *STEM image and corresponding elemental maps of vanadium, sulfur, carbon and oxygen showing the formation of vanadium sulfide particles embedded in carbon fibers. Images taken from [4].*

The project CarMON (2017 to 2020) developed new materials for lithium-ion batteries and water desalination, studied their electrochemical behaviour, and correlated the properties to their nanostructure. It was financially supported by the Leibniz Association and fostered close cooperation between the INM - Leibniz Institute for New Materials, Saarbrücken, the Leibniz Institute for Plasma Science and Technology, Greifswald, and the NG group at the MPIE. Several publications resulted from the collaboration [1-5], and several on-site meetings were conducted in Greifswald, Saarbrücken, and Düsseldorf. These two-day meetings included scientific discussion and experimental planning and were also used to mentor and promote young scientists. Further exchange of the young scientists via laboratory visits and joint experiments allowed them to enhance their knowledge on electrochemistry, materials synthesis, and advanced microstructural characterization.

Several promising processing routes were explored by the collaboration partners, such as plasma-in-liquid synthesis, electrospinning combined with thermal sulfidation, or other wet chemical approaches. The NG group investigated the nanostructure using (scanning) transmission electron microscopy (STEM), energy-dispersive X-ray spectroscopy (EDS) [1, 2], electron energy loss spectroscopy (EELS) [1, 2] and electron tomography [2].

A vanadium oxide/carbon core/shell hybrid material was synthesized via a two-step procedure involving a thermal treatment step and subsequent washing in aqueous hydrochloric acid [3]. Needle-shaped V_2O_3 nanocrystals were observed by STEM surrounded by different layered carbon species. The material was tested as anode for lithium-ion batteries and showed improved performance after 500 cycles in the potential range of 0.01-3.00 V versus Li/Li^+ [3]. This makes the hybrid attractive for future applications [3].

The material system was extended from carbon/metal oxide hybrids to sulfides as they showed promising properties. For example, the electrospinning of vanadium oxide fiber mats and subsequent thermal sulfidation using H_2S at elevated temperature resulted in an interesting hybrid composite [4]. It consisted of vanadium sulfide particles embedded within the surface region of the produced carbon fibers as revealed by EDS mapping performed in STEM mode (Fig. 1) [4]. The electrochemical properties were investigated, and it was found that the material degrades rapidly when operated between 0.1 V and 3.0 V versus Li/Li^+. However, an improved cycling stability was observed when lowering the upper voltage to 1.2 V versus Li/Li^+ [4].

Another interesting niobium disulfide/carbon hybrid material with a mesoporous structure was synthesized using a block copolymer and an organometallic niobium precursor [5]. The STEM data showed a very regular 3D network with about 20-30 nm channel size. This gyroidal morphology was beneficial for use as an electrode in lithium-ion batteries leading to a high volumetric capacity of up to 150 mAh/cm^3 [5].

References

1. Frank, A.; Dias, M.; Hieke, S. W.; Kruth, A.; Scheu, C.: Thin Solid Films 722 (2021) 138574.
2. Frank, A.; Gänsler, T.; Hieke, S. W.; Fleischmann, S.; Husmann, S.; Presser, V.; Scheu, C.: Nanoscale Adv. 3 (2021) 1067.
3. Budak, Ö.; Srimuk, P.; Tolosa, A.; Fleischmann, S.; Lee, J.; Hieke, S. W.; Frank, A.; Scheu, C.; Presser, V.: Batter. Supercaps 2(1) (2019) 74.
4. Husmann, S.; Budak, Ö.; Quade, A.; Frank, A.; Kruth, A.; Scheu, C.; Tolosa, A.; Presser, V.: J. Power Sources 450 (2020) 227674.
5. Fleischmann, S.; Dörr, T. S.; Frank, A.; Hieke, S. W.; Doblas-Jimenez, D.; Scheu, C.; de Oliveira, P. W.; Kraus, T.; Presser, V.: Batter. Supercaps 2(8) (2019) 668.

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Barrier coatings against hydrogen ingress

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The project H2BS began in January 2020 and explores the potential of ceramic thin films as barriers against hydrogen ingress of ferritic steels and iron alloys. Hydrogen levels as low as 1 part per million (ppm) in weight can induce failure of high strength steels, a phenomenon known as hydrogen embrittlement. Hydrogen diffusion barriers would enable ferritic steels for diverse applications in the hydrogen economy ranging from transportation to storage.

In this basic research project, the commercial X65 ferritic pipeline steel is chosen as a representative substrate material based on the recommendation of the project's advisory panel of industry experts. A binary Fe-Cr alloy is also chosen as a simpler model substrate. The Fe-08Cr alloy has a BCC structure, and the hydrogen diffusion and interaction in this alloy is well understood. Aluminium oxide is DC sputter deposited on the X65 and Fe-08Cr substrates. Microstructural characterization through X-ray diffraction, X-ray photoelectron spectroscopy, and (scanning) transmission electron microscopy (S)TEM, is employed to understand the microstructure of the deposited coatings. An amorphous Al_2O_3 structure with some interfacial crystallization is observed in the single layer coatings on Fe-08Cr (Fig. 1).

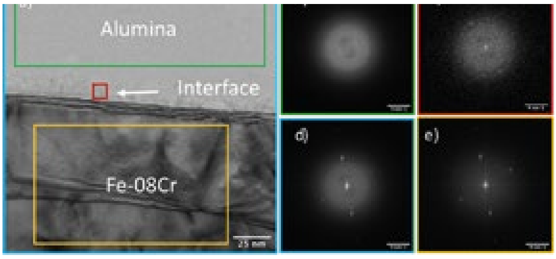

Fig. 1: *a) TEM micrograph of the film and substrate with and corresponding fast Fourier transformation data b) – e). The coating itself is amorphous while the interface shows mixed amorphous and crystalline parts.*

Hydrogen permeation and interaction in the coating is studied by Kelvin probe measurements and nanoindentation. The latter is equipped with a unique, in-house developed back-side hydrogen charging setup [1]. Rapid hydrogen diffusion through the substrate facilitates the study of coating and interfacial response to hydrogen accumulation. The breakthrough time for hydrogen charging through the substrate takes 10 min. However, with the additional coating system, no detectable signal was measured up to 17 hours. Preliminary nanoindentation and scratch testing results indicate that the coating-is resilient under hydrogen charging conditions. Further studies are underway to probe the mechanical reliability of the coating to front-side charging experiments.

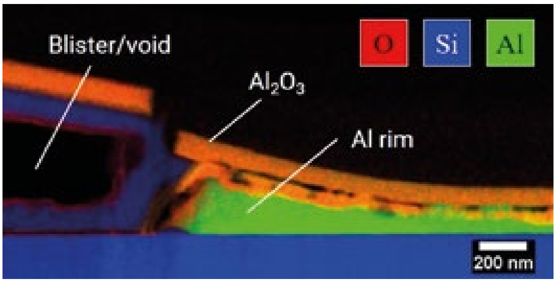

Fig. 2: *Cross-sectional STEM image overlaid with EDS elemental map reveals the microstructure change during annealing in high H_2 pressure. Blisters form with the initial amorphous Al_2O_3 layer remaining. A surrounding Al rim has built up due to dewetting. A part of the metallic film is transformed to γ-Al_2O_3.*

We also performed tests under H_2 gas pressures of 300 bar at 673 K on a bilayer film of aluminium oxide and aluminium with a total thickness of 200 nm on a silicon (100) substrate. Microstructural investigation through STEM, energy dispersive X-ray spectroscopy (EDS) and electron energy loss spectroscopy revealed changes induced by the thermal and hydrogen pressure exposure. Blisters form where the metallic aluminium was retracted by solid state dewetting, leading to ridge formation (Fig. 2). In addition, a portion of the metallic layer was transformed to γ-Al_2O_3. Comparable experiments in argon inert atmosphere at 673 K also revealed similar damage features. In combination with nanoindentation testing, we conclude that there is a modest effect of gaseous H_2 loading on the performance of the bilayer coatings.

References

1. Duarte, M.J.; Fang, X.; Rao, J.; Krieger, W.; Brinckmann, S.; Dehm, G.: J. Mater. Sci. 56 (2021) 8732.

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"PredictCorr"

N. Khayatan, M. Rohwerder

GO

PredictCorr is a joint project with the Free University of Brussels (VUB), within the framework of the Research Foundation - Flanders (FWO), in close contact with about twenty cooperating partners from industry, planned for a period of 4 years, which at the MPIE started operationally in September 2018. The motivation behind this project is that at the current state of the art, lifetime and aging assessment of coated metal are performed by means of experimental testing, combining accelerated testing and field testing, where the latter is still of crucial importance. This seriously impedes organic coating development. The only way to realise a substantial decrease in the development time of new materials is to introduce modelling in the design cycle. What is needed to realize a breakthrough in this field is a tool that can predict quantitatively and dynamically the corrosion behaviour of organic coated metals.

The scientific motivation for the project is to build a knowledge and technology platform required for achieving such a prediction of durability behaviour and estimation of lifetime of organic coated metals under long-term environmental aging and corrosion conditions. This is a very challenging task, as corrosion of an organic coated metal is the result of an intense interplay between several physical phenomena that need to be understood and modelled. The task of the MPIE will be to identify the underlying processes of the corrosion driven coating delamination and to provide experimental access to key parameters enabling us for its reliable simulation.

Over the recent years the MPIE has developed a number of techniques, such as the hydrogen permeation based potentiometric method for obtaining full I(U) curves for oxygen reduction at buried interfaces (see e.g [1]). In the reporting period substantial new insights have been obtained on the key mechanisms of corrosion driven delamination. So far, the following key processes were identified: 1) insertion of cations from the corroding defect/delaminated interface into the intact interface at the delamination front, which results in 2) a pull down of the electrode potential, which in turn 3) leads to an onset of oxygen reduction and subsequent 4) delamination. The 5) electron flow from the defect to the delaminating (and delaminated) interface that ensures steady oxygen reduction is accompanied by an according 6) cation migration along the delaminated interface. Contrary to the current common opinion that either oxygen reduction at the delamination front (for more resistant coatings) or cation migration (for weaker coatings) play the rate determining role for the progress of delamination, the results obtained so far indicate that in all cases the cation insertion at the front seems to be the kinetically limiting step. This step was found to depend on the electrode potential at the front, which in turn is affected by the ohmic drop caused by the cation migration along the delaminated interface. This ohmic drop in turn is caused by the degree of delamination at the interface, i.e. it depends on the remaining resistance against cationic mobility.

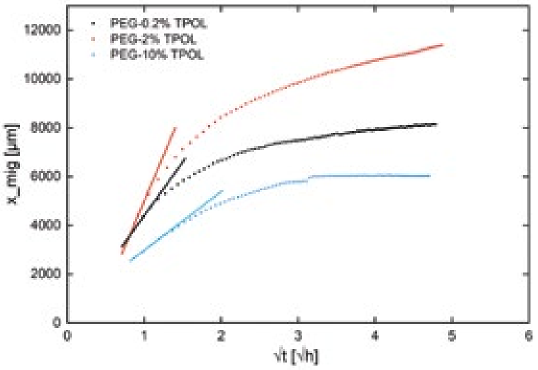

Fig. 1: *Progress of migration in humid nitrogen atmosphere for polyethylene glycol (PEG) coatings with different additions of TPOL: (2,4,6-(trimethylbenzoyl) diphenylphosphine oxide) initiator. Initially the migration rate is similar to the one of delamination (full lines).*

Thus, a quite complex process unfolds and the key idea at the MPIE was originally to investigate the underlying steps 1)-6) separately. However, it proved difficult to single out the different individual steps and to investigate them independently. For instance, it was assumed, based on earlier work, that migration at the interface could be investigated without being affected by oxygen reduction in an inert humid nitrogen atmosphere. However, it was found that migration seems to be critically affected by adsorbing species from air. In many cases the cation migration in nitrogen, which was found to be initially often at a similar rate as delamination in air, was slowing down to a halt after a few hours (e.g. Fig.1). Whether this is linked to desorption of initially adsorbed CO_2, which we found to play an important role in migration [2], or even adsorbed O_2 (see p. 125), has yet to be elucidated.

Hence, the main focus so far was on the main rate determining step, i.e. the cation insertion at the front, where significant progress could be made.

References

1. Zhong, X. K.; Schulz, M.; Wu, C. H.; Rabe, M.; Erbe, A.; Rohwerder, M.: Chemelectrochem. 8(4) (2021) 712.
2. Shkirskiy, V.; Uebel, M.; Maltseva, A.; Lefèvre, G.; Volovitch, P.; and Rohwerder, M.: npj Mater. Degrad. 3 (2019) 2.

Cluster of Excellence “Ruhr Explores Solvation”

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[1]GO, [2]CM

The Cluster of Excellence RESOLV (Ruhr Explores Solvation Science) was established in November 2012 and the second funding phase is running since 2019. It is an interdisciplinary research cluster co-hosted at Ruhr-Universität Bochum (RUB) and TU Dortmund. It involves partners from several departments from RUB, groups from TU Dortmund, University Duisburg-Essen, Fraunhofer UMSICHT Oberhausen, MPI für Kohlenforschung, MPI for Chemical Energy Conversion and the MPIE. From these institutions, currently more than 200 scientists (professors, independent researchers, postdocs and PhDs) are enlisted in RESOLV [3].

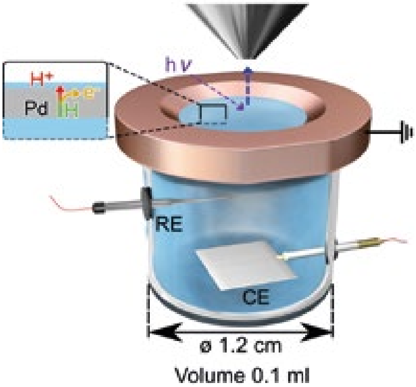

Fig. 1: *Experimental setup for an electrode in the dry for measurements with near ambient pressure x-ray photoelectron spectroscopy (XPS). The metal electrode is electrochemically charged with atomic hydrogen on the entry side. The hydrogen diffuses and sets a distinct potential at the dry, exit side where an ultrathin electrolyte film covers the interface.*

The MPIE departments GO and CM contribute with their strong expertise in surface science, electrochemistry and *ab initio* modelling to the activities of RESOLV, currently focussing in research area I: “Local Solvent Fluctuations in Heterogenous Systems”. To that end in 2020 the joint MPIE-RUB early career research group: “Spectroscopy at Electrochemical Interfaces” was established in the GO department. The group uses interface spectroscopic methods to study solvation and other dynamic effects at interfaces (see p. 45). The specific properties of water near interfaces are studied mainly using vibrational spectroscopic methods, which yield information about the strength of the hydrogen-bonding network of water. In a recently started project, the local structure of thin water layers on organic nanostructured self-assembled monolayers with tuneable surface chemistry will be studied by local vibrational spectroscopy with nanometric resolution (AFM-IR).

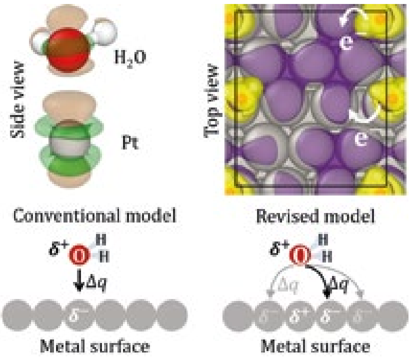

Fig. 2: *Our ab initio molecular dynamics calculations led to a revision of the well-established picture of water-metal bonding. The electron difference density (top) for the Pt(111)/water interface reveals the involvement of Pt atoms neighbouring the top water adsorption site (right). As consequence the number of available surface adsorption sites reduces. It also shows that the chemisorbed water molecules become metallic [2].*

Further research in the GO department, closely related to RESOLV is the so-called „electrode in the dry“. We showed how to use this concept e.g. for detecting hydrogen in materials and performed permeation measurements with high local resolution and high sensitivity. Recently, this approach was applied to measure full current-potential curves of oxygen reduction on a dry palladium surface [1], covered with just ultra-thin electrolyte layers in the sub-nanometre range. Currently, the “electrodes in the dry” are studied as a powerful tool for an in-depth characterization of electrochemical double layers and it is explored how the concept can be transferred to other electrode materials such as iron(oxide), iridium and gold.

Two projects within the CM department closely related to RESOLV activities explore fundamental questions of surface solvation and interface reactions. One project, carried out in close collaboration with experimental colleagues from the University of Duisburg-Essen, focuses on low index Pt surfaces in contact with water exploring the interface geometric and electronic structure and the interplay between water and hydrogen adsorption. The second project, aims at understanding charge transfer reactions at the solid/water interface.

References

1. Zhong, X.; Schulz (née Uebel), M.; Wu, C.-H.; Rabe, M.; Erbe, A.; Rohwerder, M.: Chem. Electro. Chem. 8 (2021) 712.
2. Surendralal, S; Todorova, M.; Neugebauer, J.: Phys. Rev. Lett. 126 (2021) 166802.
3. https://solvation.de; https://www.fp-resomus.ethz.ch/

Gas turbines of tomorrow: from atoms to turbine blades

P. Kontis, S. Makineni, J. He, S. Katnagallu, X. Wu, A. Saksena, J. Mianroodi, D. Raabe, B. Gault

MA

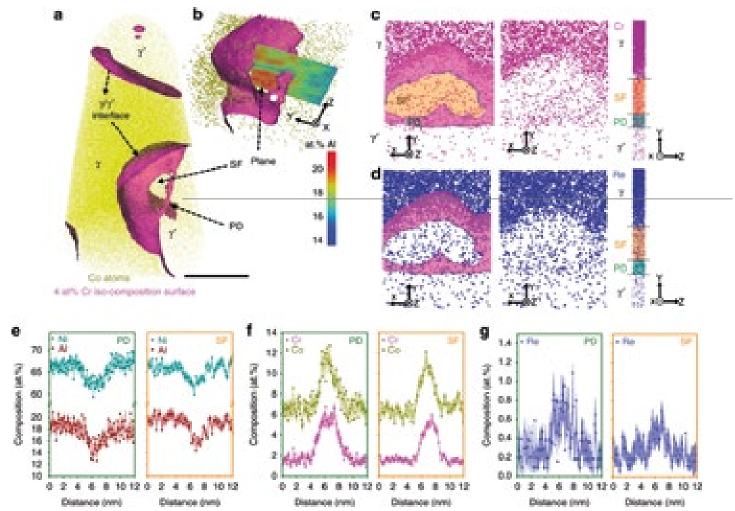

Fig. 1: *An APT reconstruction and analyses of the Re interactions with structural defects in a creep-deformed Ni-based alloy developed as part of the SFB TR103 (from [5], for details, see caption in the article).*

The atom probe tomography (APT) group in the MA department was an integral part of the second phase of the SFB/TR 103 “From atoms to turbine blades – a scientific basis for a new generation of single crystal superalloys”, which ended in December 2019. The joint research centre with the Ruhr-Universität Bochum and Friedrich-Alexander-Universität Erlangen-Nürnberg entered its third phase in January 2020 and is funded by the German Research Foundation (DFG).

We developed correlative protocols involving high-end scanning and transmission electron microscopy and atom probe tomography during the second phase [1]. These were then used to study the details of the segregation of solutes to structural defects in a range of Ni- and Co-based superalloys deformed at high temperature during the second phase. For instance, we showed that diffusion of solutes along crystal defects controls the formation of micro-twins during creep deformation of a Co-base superalloy [3]. The formation of micro-twins is undesirable for the mechanical performance of superalloys. Our results, provided new insights into a solute diffusion mechanism occurring during micro-twinning, that can be exploited during the design of new advanced Co-based superalloys, by limiting the formation of micro-twins.

Towards the end of the second phase, we focused on the behaviour of Re, one of the key elements for creep resistant alloys. First, we revealed Re-segregation to dislocations in a creep-deformed model binary NiRe by using the newly-developed approach of analytical field-ion microscopy, and rationalised this through atomistic simulations [3]. Second, we targeted the low-angle boundaries present in a single-crystal Ni-based superalloy containing Re, and revealed Re segregating to the dislocations forming the boundary [4].

Finally, we measured Re segregated to dislocations and stacking faults, along with other solutes. We demonstrated that the local Re concentration at defects varies over the course of the creep deformation process. The creep strain rate fluctuates depending on the stage of the creep process, and as a consequence, the dislocation velocity varies throughout the creep deformation. We found less Re segregated as the dislocation velocity, and hence creep strain rate, increases. Our experimental observations have been further supported by atomistic phase field simulations [5].

Overall, our investigations provide new insights at the near-atomic level, into processes taking place during the deformation of superalloys. This fundamental understanding can pave the way to design materials, which can withstand higher operational temperatures, leading eventually to more efficient and environmentally friendly aero-engines.

References

1. Makineni, S.K.; Lenz, M.; Kontis, P.; Li, Z.; Kumar, A.; Felfer, P.J.; Neumeier, S.; Herbig, M.; Spiecker, E.; Raabe, D.; Gault, B.: JOM 70 (2018) 1736.
2. He, J.; Zenk, C.H.; Zhou, X.; Neumeier, S.; Raabe, D.; Gault, B.; Makineni, S.K.: Acta Mater. 184 (2020) 86.
3. Katnagallu, S.S.; Stephenson, L.T.; Mouton, I.; Freysoldt, C.; Subramanyam, A.P.A.; Jenke, J.; Ladines, A.N.C.; Neumeier, S.; Hammerschmidt, T.; Drautz, R.; Neugebauer, J.; Vurpillot, F.; Raabe, D.; Gault, B.: New J. Phys. 12 (2019) 21.
4. He, J.; Scholz, F.; Horst, O. M.; Thome, P.; Frenzel, J.; Eggeler, G.; Gault, B.: Scr. Mater. 185 (2020) 88.
5. Wu, X.; Makineni, S.K.; Liebscher, C.H.; Dehm, G.; Mianroodi, J.R.; Shanthraj, P.; Svendsen, B.; Bürger, D.; Eggeler, G.; Raabe, D.; Gault, B.: Nat. Comms. 11 (2020) 389.

Atomic scale redistribution of carbon during the transformation from austenite to martensite in steels

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The mission of this joint project is to reveal the "Atomic scale redistribution of **C**arbon during the **TR**ansformation from **A**ustenite to **M**artensite in steels" (C-TRAM). It is driven, on the one hand, by the applicational relevance of martensitic steels due to their extraordinary strength and the adjustability of their toughness and formability over a wide range by simple technological treatments. On the other hand, the nonequilibrium distribution of C atoms in a transforming microstructure is a scientifically exciting topic.

Both aspects are connected: C atoms are super-saturated in freshly formed martensite and their concentration and distribution is decisive for the strength and toughness of the martensite. The distribution (and potential ordering) of carbon atoms happens at the transformation front, the austenite-martensite interface. These processes, and in particular the underlying kinetics of the C diffusion and the interface mobility are not yet understood in detail down to the atomic scale, despite their high relevance for the design of martensitic steels with tailored mechanical properties. Both, theoretical and experimental investigations are so far restricted to either idealized material systems or limited resolution of joint structural and chemical data.

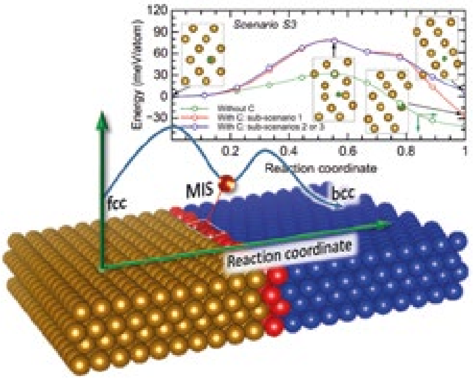

Fig. 1: *Ab initio determined minimum-energy path and impact of C on the structural evolution from a metastable intermediate structure (MIS) to the martensite. Figure adapted from [1].*

Within this project we address these open questions by combining high resolution theoretical and experimental investigations in a complementary approach for Fe-TM-C steels. This is achieved by a strong team of partners from France and Germany. On the theoretical side, the expertise of the group of T. Hickel in *ab initio* thermodynamics (see p. 71) and structural phase transformations (Fig. 1), will be combined with *ab initio* based kinetic simulations performed primarily by the group of P. Maugis as well as atomic density field theory and quasiparticle approaches performed by the group of H. Zapolsky. On the experimental side, atom probe tomography (APT) performed in the group of F. Danoix and (high resolution) transmission electron microscopy (TEM) in the group of S. Sandlöbes-Haut will be used to combine structural and chemical data. Eventually, correlative TEM-APT analyses to achieve fully correlative data on the local structure and chemistry will be applied for selected key samples (Fig. 2).

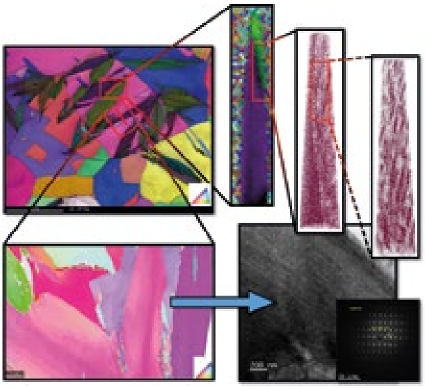

Fig. 2: *Correlative study TEM-APT analysis of a Fe-C microstructure containing austenite and martensite performed at RWTH Aachen University.*

The high significance of this project for the MPIE goes well beyond the microstructure formation in an important class of steels. The focus on the martensite-austenite interface involves not only the analysis of the local atomic interface structures, but also possibly formed interfacial states during C redistribution. Defect phases are, for example, central for the CRC 1394 (see p. 76). Central project topics like the structural and chemical distribution of C, the order-disorder transition of C atoms, the formation of carbides, and the formation of reverted austenite further strengthen the links between the CM and MA department. In particular B. Gault was strongly involved when starting this exciting scientific journey.

References

1. Zhang, X.; Hickel, T.; Rogal, J.; Neugebauer, J.: Phys. Rev. B 942 (2016) 104109.

Experiments for optimised CALPHAD modelling

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SN

Within the framework of the European research programme Clean Sky 2 the project ADVANCE is devoted to improve an existing CALPHAD database for the development of more creep and corrosion resistant γ TiAl-based alloys. These alloys are needed for the design of lightweight, less fuel-consuming aero engines to reduce CO_2 and NO_x emissions and aircraft noise.

The ADVANCE project idea has been put forward by MTU Aero Engines AG, Munich, Germany, who also acts as topic manager for the project. The MPIE is part of the international consortium that has been successful in winning the competitive call. At the MPIE the majority of the experiments including production of about 100 different alloys under high-purity conditions is performed. Thermo-Calc Software AB, Sweden is compiling the final database, performs modelling for checking the consistency of the data and coordinates the consortium, while Helmholtz-Zentrum Hereon, Geesthacht, Germany performs *in situ* high-energy X-ray diffraction using synchrotron radiation and Montanuniversität Leoben, Austria executes supplementary transmission electron microscopy (TEM) and atom probe tomography (APT) studies.

Within the project, the Ti-Al side of the systems Ti–Al–X–Y with X = Nb, Mo, W, O, B, Zr, C, Si and X/Y = Nb/Mo, Nb/W is studied at temperatures ranging between 600-1300 °C. For these systems data for phase equilibria at individual temperatures are either missing or doubtful, and phase transformations in dependence on temperature may not have been settled. Though at least part of these systems have been repeatedly investigated, data are still missing or contradictory due to the experimental difficulties associated with these systems.

At the MPIE, large rods of high-purity alloys are produced by levitation melting or using an advanced arc melter. Compositions, impurity contents, and homogeneity are established by wet-chemical analysis and electron probe microanalysis (EPMA) of larger areas. Heat treatments are either performed by encapsulating the samples or using a double-crucible technique. Both result in a minimum uptake of impurities, which is checked again by wet-chemical analysis. Investigations on alloys of fixed composition are complemented by analysing diffusion couples to establish phase equilibria at lower temperatures. After quenching, microstructures are analysed by light-optical microscopy and scanning electron microscope. The phase content is determined by X-ray diffraction and compositions of coexisting phases are established by EPMA. Temperatures of phase transformations are determined by differential thermal analysis, employing sequences of different heating rates. All evaluated data are finally used to establish up to seven isothermal sections for each investigated alloy system and additional vertical sections. Besides experimental work, critical assessment of existing data is also performed [1].

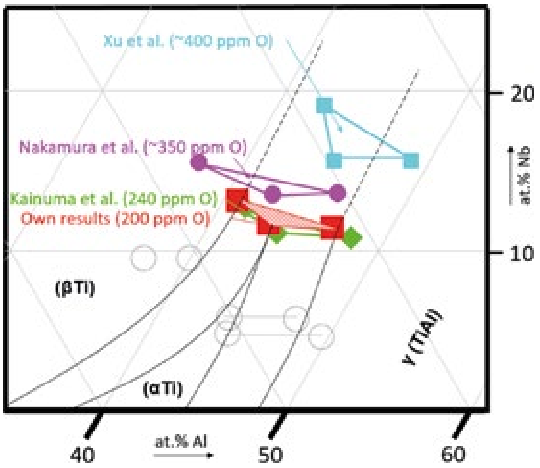

Fig. 1: *Partial isothermal section of the Ti–Al–Nb system at 1300 °C with experimental results from the literature and own results regarding the oxygen-related shift of the tie-triangle (αTi)+(βTi)+γ (TiAl) [2].*

For the development of creep resistant γ TiAl-based alloys, Ti–Al–Nb is a key system and respective commercially available materials are known as TNB alloys. A critical issue is that oxygen has a significant effect on phase equilibria, shifting the phase compositions and, thereby, changing the microstructure and thus the properties of the alloys. Fig. 1 shows that at 1300 °C already an increase from 200 to 400 wt. ppm O results in a considerable shift of the (αTi) + (βTi) + γ (TiAl) tie-triangle towards higher Nb contents. As an uptake of oxygen during industrial processing and application is unavoidable, the knowledge of the influence of oxygen impurities on phase equilibria is essential for any aimed alloy development.

More details about ADVANCE can be found at https://thermocalc.com/advance

References

1. Kahrobaee, Z.; Palm, M.: J. Phase Equilib. Diffus. 41 (2020) 687.
2. Distl, B.; Dehm, G.; Stein, F: Z. Anorg. Allg. Chem. 646 (2020) 1151.

Combinatorial metallurgy and processing

H. Springer

MA, now also at: Institute of Metal Forming, RWTH Aachen University, Germany

The collaborative research center (CRC) 1232, coordinated by the University of Bremen, was concerned with the development of a novel high throughput method for the development of metallic structural materials. The overall aim was to contribute to the efficient and focused identification of alloy compositions and processing chains for materials with a specific performance profile. The method – termed „Farbige Zustände" or "coloured states" – combined novel processes for the primary shaping (especially small droplets), microstructure formation ("coloration") and characterization of microscopic material samples with sample logistics as well as mathematical and computer science based methods for the analyses of large amounts of data. The goal was to achieve the transformation of determined descriptors towards macroscopic material properties of the performance profile by a heuristic predictor function. The method „Farbige Zustände" aims at a paradigm shift in the development of structural materials towards a resource efficient high throughput method for the experimental exploration of novel groups of materials [1].

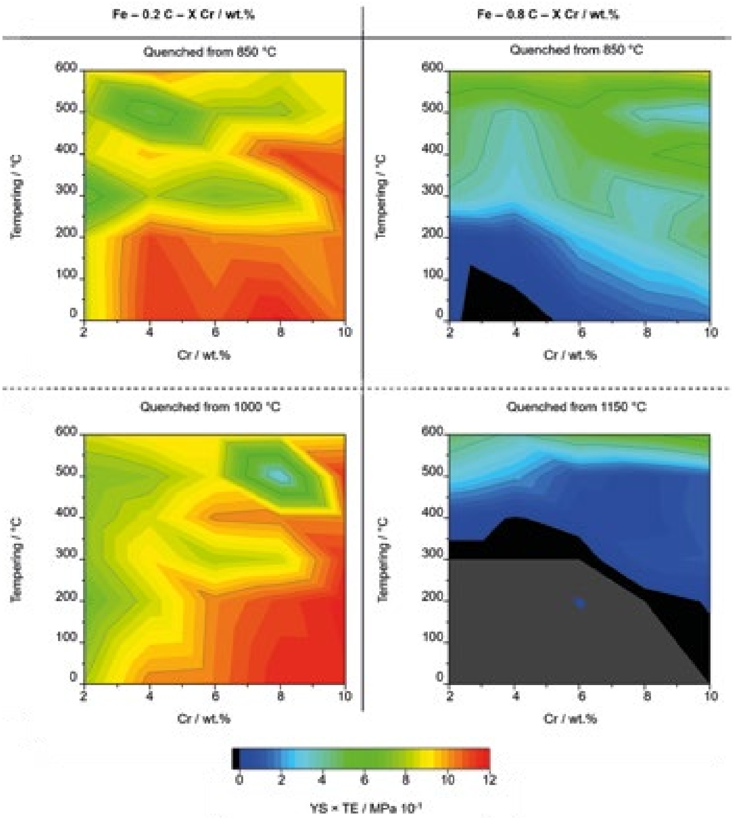

Fig. 1: *Heat maps displaying the property profiles of 100 states of Fe-Cr-C steels investigated with the combinatorial RAP methodology [4].*

In the first funding period of the CRC 1232 starting in 2016, the MPIE was involved with the project "correlative study towards experimental validation of the high throughput methodology", which had two main objectives. The first was to provide the CRC with 70 kg batches of conventionally produced Fe-Cr-C alloys serving as "supports" to validate and calibrate the high throughput methodology based on small metallic droplets [1, 2]. The second objective was to investigate the property profiles of yet unexplored regions of the Fe-Cr-C system (Fig. 1). By means of the combinatorial rapid alloy prototyping (RAP) approach developed at the MPIE [3], materials in a wide range of chromium and carbon concentrations were investigated. The resultant property profiles of 100 "coloured" states (Fig. 2) were supplied to the CRC for developing the predictor function [4].

Due to the start of his Heisenberg-professorship at the RWTH Aachen University, H. Springer left the CRC 1232 at the end of the first funding period. Further participation of the MPIE was planned by E.A. Jägle with a focus on additive manufacturing of aluminium alloys, but funding of the initiative was not continued. Nevertheless, the collaboration established by the CRC between the MPIE and the Leibniz Institut IWT spawned a joint research into the development of high modulus steels [5], which is continued in a project on additive manufacturing of these novel lightweight design materials funded by the German Federation of Industrial Research Associations (AiF).

References

1. https://www.uni-bremen.de/farbige-zustaende.de
2. Sonnenberg, H.; Clausen, B.: Materials 13 (2020).
3. Springer, H.; Raabe, D.: Acta Mater. 60 (2012) 4950.
4. Baron, C.; Springer, H.: steel res. int. (2019) 1900404.
5. Springer, H.; Baron, C.; Szczepaniak, A.; Uhlenwinkel, V.; Raabe, D.: Sci. Rep. 7 (2017) 2757.

Damage controlled forming processes

C. Tian[1], L. Christiansen[1], D. Ponge[2], G. Dehm[1], C. Kirchlechner[1,3]

[1]SN, [2] MA, [3]Karlsruhe Institute of Technology (KIT)

Damage is not failure! But materials used in any technological application contain damaged sites. This is very obvious for forming processes, where voids or micro/nano-cracks frequently occur in metallic components. They can deteriorate mechanical properties such as component stiffness, strength and fracture toughness, finally influencing the application performance and lifetime of products. However, in traditional processing routes, the damage generated by forming is not quantitatively considered and investigated. A common counter measure is increasing the component thickness to accommodate the uncertainty of safety potential, which goes against the light weight and sustainability trend. Therefore, this project aims at damage-predictable and -controllable processing routes to fully exploit the materials' service potential and deal with the sustainability issue.

The research of the Transregio 188 bridges from microscale regimes up to meters and is divided into three topics: forming processes, modelling and materials characterization. The collaboration partners are RWTH Aachen University, Technical University Dortmund, Brandenburg University of Technology, MPIE and KIT. At the MPIE, we are engaged in resolving the local mechanical properties of the constituent phases ferrite and martensite in two dual phase DP800 steels, and in physically understanding the damage initiation from a micromechanical perspective.

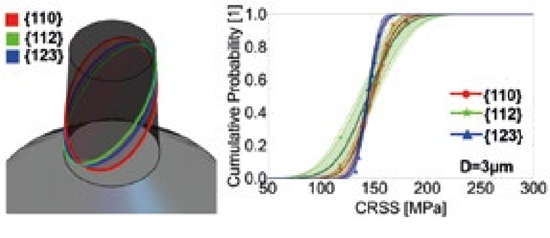

Fig. 1: *A Mathematica drawn ferrite pillar according to the electron backscatter diffraction (EBSD) orientation showing different slip traces distinguished by colour (left). Cumulative distribution of CRSS for slip plane families {110}, {112}, {123} (right) [1].*

Through pillar compression tests, we are capable of investigating the plasticity of constituent phases, for instance, identifying activated slip systems and respective critical resolved shear stress (CRSS), as shown in Fig. 1. By microcantilever bending as in Fig. 2, we can extract fracture initiation toughness of damage sensitive sites, particularly martensite islands in DP steels. Further, we apply such characterization techniques as ECCI (electron channelling contrast imaging) and APT (atom probe tomography) to resolve the influence of microstructure and chemical composition on the resulting mechanical properties.

According to the research work of the first funding period, ferrite activates dislocation slip on {110}, {112}, {123} plane families following Schmid's law. The CRSS of the three slip plane systems is found to be very similar with values of approximately 150 MPa for 2 µm sized micropillars. Martensite islands, absorbing most of carbon with roughly 3 at. % C, show a compressive yield strength up to 3 GPa. A huge mechanical heterogeneity between the two phases is quantitatively determined [1-2]. Martensite islands initiate fracture in an elastic plastic fashion [3-4].

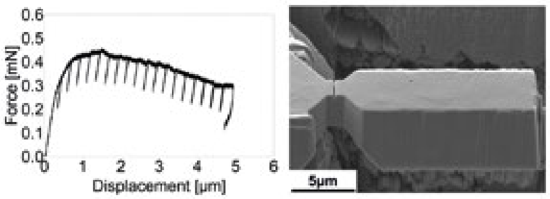

Fig. 2: *A representative force displacement curve for a beam showing crack growth during the cantilever bending test (left). Necked beam geometry contains martensite at the neck region (right). Martensite in this DP grade has a carbon content approximately 3 at.%. J-integral curve can be extracted [3].*

All these findings not only serve as input parameters for crystal plasticity finite element modelling for our cooperation partners, but also contribute to a deeper understanding of damage initiation behaviour in dual phase steels. This topic will be further studied in the second funding period (2021 – 2024) and supported by novel electrical resistivity approaches to sense damage.

References

1. Tian, C.; Ponge, D.; Christiansen. L.; Kirchlechner, C.: Acta Mater. 183 (2020) 274.
2. Tian, C.; Dehm, G.; Kirchlechner, C.: Materialia 15 (2021) 100983.
3. Tian, C.; Kirchlechner, C.: J. Mater. Res. 36 (2021) 2495.
4. Kusche, C.F., Dunlap, A.; Pütz, F.; Tian, C.; Kirchlechner, C.; Aretz, A.; Schwedt, A.; Al-Samman, T.; Münstermann, S.; Korte-Kerzel, S.: Prod. Eng. 14.1 (2020) 95.

Non-destructive tomography with sub-micrometre resolution

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[1]SN, Karlsruhe Institute of Technology, Karlsruhe, Germany, [3]CRG-IF BM32 at ESRF, Grenoble, France

The XmicroFatigue project, funded by the German and French Research Foundations (DFG-ANR), aims at developing a novel microscope for interlinking the mechanical properties and the underlying and evolving defect structure in three dimensions, non-destructively with sub-micrometre resolution. The microscope is primarily used for investigating the fatigue damage evolution at a single grain boundary during cyclic bending experiments.

Why do we need such a tool? Advanced characterization techniques, like scanning electron microscopy (SEM) or transmission electron microscopy (TEM), are either surface sensitive or require an electron transparent thin foil. Surface effects in dislocation mechanics, for instance the presence of image forces, cause dislocation annihilation at the sample surface, which significantly modifies the observed material behaviour. Non-destructive synchrotron-based techniques offer a penetration depth exceeding the mean free path of dislocations in advanced microstructures considerable, and therefore allow for monitoring the "real" mechanical response of materials at the micron scale.

XmicroFatigue is based on a differential aperture x-ray microscopy (DAXM) [1], which is combined with a load frame being able to deform micron-sized samples in displacement-controlled mode [2]. The microscope is installed at our partner institute CEA Grenoble, which is operating the beamline BM32 at the ESRF synchrotron. A Laue pattern originating from a 1x1x1µm³ sized voxel can be calculated from partially shadowed diffraction patterns. Subsequently, the sample orientation, the density of geometrically necessary dislocations (GNDs) and the deviatoric strain tensor can be mapped in 3D tomographically.

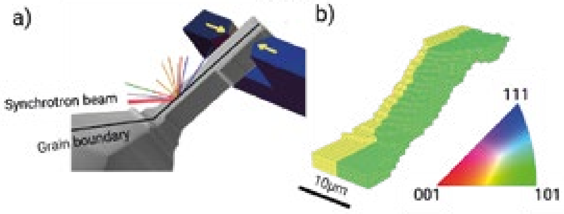

Fig. 1: *a) Schematic of the micro fatigue experiment on a single grain boundary. b) Reconstruction of the grain shape and grain orientation.*

The microscope was successfully applied to analyse the microstructural changes in the vicinity of a grain boundary, which was located at the neutral plane of a focussed ion beam (FIB) milled copper microcantilever. The sample was cyclically bent with 3D tomograms before testing, after ¼, ½, ¾ and a full cycle. A sketch of the experimental setup as well as a reconstruction of the crystal orientation is shown in Fig. 1. The two grains (separated by the grain boundary) can clearly be seen in yellow and green. Each cuboid represents a voxel with a size of 1x1x1 µm³.

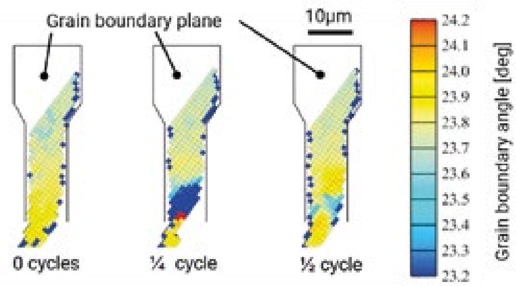

Fig. 2: *Grain boundary angle before loading, after ¼ and ½ cycles. Note the significant change in grain boundary angle after ¼ cycle, which is partly reduced during back-bending.*

Our experiments show significant cyclic slip-irreversibility during fatigue loading: before loading (0 cycles), the grain boundary angle is 23.7 to 23.9°. As soon as the sample is bent in one direction (1/4 cycles), GNDs are not only stored in the grain interior but also enter the grain boundary, resulting in a local drop of the grain boundary angle to below 23° (blue region Fig. 2). After straightening the cantilever, most of the GNDs previously stored at the grain boundary leave the grain boundary. However, in some locations (light blue area) fatigue damage slowly accumulates and GNDs are cyclically stored at the boundary.

The equipment developed within XmicroFatigue was also successfully used for coherent diffraction [3] experiments and to investigate the orientation spread in nonporous gold [4]. The project was transferred to the Karlsruhe Institute of Technology due to the appointment of C. Kirchlechner in January 2020.

References

1. Larson, B.C., Yang, W., Ice, G.E., Budai, J.D., Tischler. J.Z.: Nature 415 (2002) 887.
2. Kirchlechner, C., Keckes, J., Micha, J.-S., Dehm, G.: Adv. Eng. Mat. 13 (2011) 8.
3. Richard, M.-I., Cornelius, T.W., Lauraux, F., Molin, J.-B., Kirchlechner, C., Leake S.J., Carnis, J., Schülli, T.U., Thilly, L., Thomas, O.: Small 16 (2020) 1905990.
4. Elder, K.L.M., Beck Andrews, W., Ziehmer, M., Mameka, N., Kirchlechner, C., Davydok, A., Micha, J.-S., Chadwick, A.F., Lilleodden, E.T., Thornton, K., Voorhees, P.E.: PNAS 118 (2021) 30.

Optical manipulation and coherent control of low-dimensional electronic systems

A. S. Razzaq, S. Wippermann

GO

Emerging energy conversion, storage and energy-efficient computing technologies critically depend on understanding and controlling the precise mechanistic details of chemical reactions and their dynamics at the femtosecond time-scale. Based on correlated experimental and simulation techniques - a central focus at the MPIE - the German Research Foundation (DFG) FOR 1700 research group concentrates on the exploration, manipulation and control of one-dimensional (1D) electronic materials as well-defined model systems in this context. It fosters networking activities between the universities of Hannover, Würzburg, Paderborn, Osnabrück, Duisburg-Essen and TU Berlin, as well as the ISAS Berlin, the FHI Berlin and MPIE. In the current running period, additional collaborations have been established with the MPI for Biophysical Systems, Göttingen, in conjunction with the SFB 1073 – "Atomic scale control of energy conversion", and the IBS Center for "Low Dimensional Electronic Systems", Pohang.

FOR 1700 particularly targets surface-supported atomic wires, featuring fascinating fundamental physical properties, such as electronic instabilities with associated phase transitions, charge-density wave (CDW) formation and non-Fermi liquid behaviour. The Si(111)-(4x1)In surface is one of the most widely investigated systems in this context. It exhibits a reversible metal-insulator transition into a CDW ground state with (8x2) translational symmetry at temperatures below T_C = 120 K. This transition is of 1st order, hampered by an energy barrier. We demonstrated that the insulating and the metallic phases are able to coexist at temperatures close to T_C, giving rise to a phenomenon called electronic phase separation (EPS) and the formation of novel types of interfaces, such as metal-CDW junctions [1]. The transition itself is driven by multiple soft phonons with a strong coupling between electronic and lattice degrees of freedom. Different superpositions of these phonons transform the wires into structurally distinct but energetically degenerate CDW states. As a consequence, the CDW phase supports fundamental excitations that take the form of interfaces between these degenerate states, and that feature topological properties with associated chiral edge states inside the CDW band gap.

For all these reasons, atomic wires are promising targets in order to develop and test coherent optical control schemes inspired by concepts from femtochemistry. We design suitable control schemes and explore how intense light fields and femtosecond laser pulse sequences can be used to guide the nuclear and electronic degrees of freedom through their complex multi-dimensional potential energy landscape into new states of matter. Fig. 1 shows an example at low temperatures, where a specific CDW state is targeted with a fs laserpulse sequence and optically switched into a supercooled metallic state.

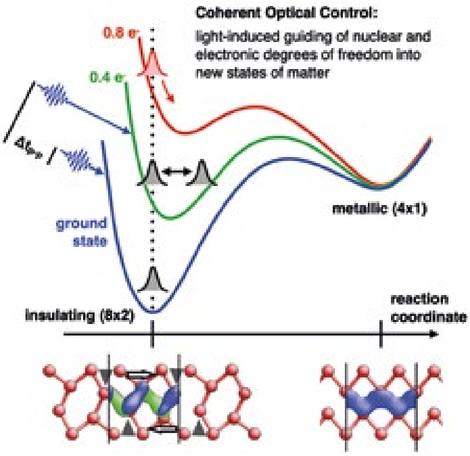

Fig. 1: *Ground and excited-state potential energy surfaces (PES) along a reaction coordinate, that transforms surface-supported indium atomic wires in an insulating CDW state into a supercooled metallic phase. Schematic structural models are shown below the PES, where red balls indicate indium atoms. An optical transition between the filled/empty electronic orbitals marked in blue/green, respectively, excites a displacive shear motion, indicated by white arrows, and thereby creates coherent oscillations of the wire structure along the reaction coordinate. A subsequent optical pulse with a carefully timed delay* $\Delta t_{p\text{-}p}$ *selectively drives the system over the reaction barrier (in-phase excitation) or de-excites the system (anti-phase exc.).*

Our research activities focus on understanding outstanding fundamental open questions in the context of low dimensional electronic systems and benefit from mutual insights obtained in different areas of interface chemistry and surface science research at the MPIE. In order to foster exchange at an international level, in February 2019 we conducted a three-day workshop at Ringberg Castle on "Surface Science: The Past, Present and Future", in order to bring together leading scientists from the areas of low dimensional electronic systems, surface spectroscopy and strongly correlated systems.

On-going research explores how to further extend our coherent control scheme to achieve mode-selective control over the individual soft phonon modes that drive the various structural and electronic transitions inherent to low-dimensional electronic systems.

References

1. Song, S. K.; Samad, A.; Wippermann, S.; Yeom, H. W.: Nano Lett. 19 (2019) 5769.

Hysteresis design of magnetic materials for efficient energy conversion

B. Gault, D. Palanisamy, K. Schweinar, N. Polin, Z. Rao

MA

Soft and hard magnetic materials are at the core of technologies for power generation, for instance in wind turbines, power conversion in transformers, and electric vehicles. Enhancing the performance of these devices is critical to reducing carbon emissions worldwide by improving energy conversion efficiency and by weight-reduction of devices to limit the ecological impact. These are among the technologies that need sustainable (i.e. limiting the amount of critical elements), low-cost magnetic materials, which, ideally, have magnetic hysteresis properties that can be tailored.

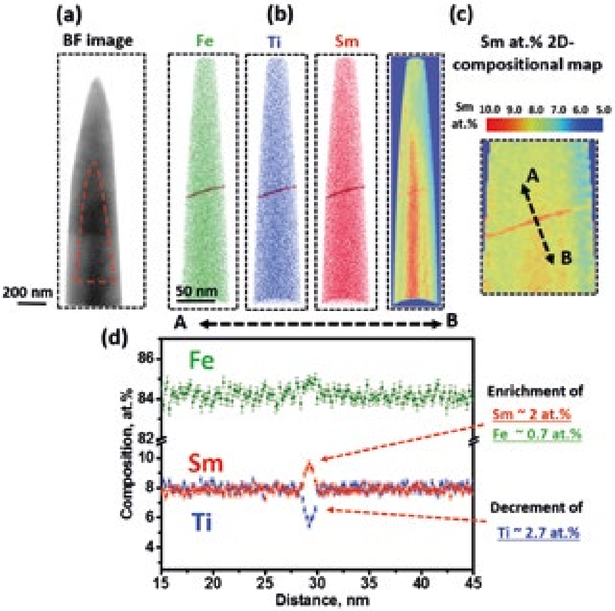

Fig. 1: *(a) Bright-field (BF) image of one of the investigated APT specimens. (b) Atom probe tomography reconstruction of the elemental distribution of Sm, Fe, and Ti. (c) 2D compositional map of Sm and (d) chemical composition distribution at the twin boundary region.*

Magnetic properties are, in part, underpinned by the material's microstructure and its composition, yet much remains unknown of the relationships linking these. As the importance of magnetic materials is increasing worldwide, interest was growing across the MA department to expand our research further into magnetic materials [1]. We then initiated a collaboration around the analysis of magnetic materials by correlative transmission electron microscopy / atom probe tomography with O. Gutfleisch from TU Darmstadt, a world-leading expert [3, 4, 5]. We managed to measure the local composition at twin boundaries that help explain the magnetic properties of 1:12 compounds, and demonstrated the importance of the phase organisation and composition near grain boundaries in pinning-type magnets, by an original approach combining isotope-labelled hydrogen and atom probe tomography.

O. Gutfleisch then invited to join forces and apply to the German Research Foundation (DFG) for a collaborative research centre joining TU Darmstadt and the University Duisburg-Essen. The first phase of this Collaborative Research Centre (SFB) TR270 started in January 2020. He also now leads the De Magnete group at the MPIE (see p. 25). The key motivation for this SFB TR is to advance the understanding of the complex interplay between local phase morphology and composition, structural defects, and the hysteresis and magnetic properties of bulk magnetic materials.

A scientist in the atom probe group now performs primarily atom probe tomography on novel materials developed across the collaborative research centre, mostly targeting hard magnets. Based on these insights, we can establish the foundation for designing novel, sustainable and efficient permanent materials. In parallel, we are also contributing to the SFB by developing machine-learning based data processing techniques facilitating the analysis of these bulk permanent magnets [5].

References

1. Palanisamy, D.; Kovács, A.; Hegde, O.; Dunin-Borkowski, R. E.; Raabe, D.; Hickel, T.; Gault, B.: Phys. Rev. Mater. 5 (6) (2021) 064403.
2. Palanisamy, D.; Ener, S.; Maccari, F.; Schäfer, L.; Skokov, K. P.; Gutfleisch, O.; Raabe, D.; Gault, B.: Phys. Rev. Mater. 4 (5) (2020) 054404.
3. Ener, S.; Skokov, K. P.; Palanisamy, D.; Devillers, T.; Fischbacher, J.; Eslava, G. G.; Maccari, F.; Schäfer, L.; Diop, L. V. B.; Radulov, I.; Gault, B.; Hrkac, G.; Dempsey, N. M.; Schrefl, T.; Raabe, D.; Gutfleisch, O.: Acta Mater. 214 (2021) 116968.
4. Ye, X.; Yan, F.; Schäfer, L.; Wang, D.; Geßwein, H.; Wang, W.; Chellali, M. R.; Stephenson, L. T.; Skokov, K.; Gutfleisch, O.; Raabe, D.; Hahn, H.; Gault, B.; Kruk, R.: Adv. Mater. 33 (5) (2021) 2006853.
5. Wei, Y.; Varanasi, R. S.; Schwarz, T.; Gomell, L.; Zhao, H.; Larson, D. J.; Sun, B.; Liu, G.; Chen, H.; Raabe, D.; Gault, B.: Patterns 2 (2) (2021) 100192.

Research funding

K. Hübel

Research Coordination Office

Professional support in research funding

The major goal of providing professional support in research funding is to increase the career prospectives of our scientists. K. Hübel is providing expert advice on international, European and national funding opportunities. She gave advice on all kinds of funding from individual (e.g. Humboldt Research Fellowship for Postdoctoral Researchers, Marie Skłodowska-Curie Individual Fellowships or ERC Grants) to project funding (e.g. Horizon 2020, German Ministry for Science & Education or German Research Foundation). Furthermore, special attention was drawn to prizes and awards.

Competitive applications

Professional and administrative assistance was provided for researchers throughout the whole process of application. Support was given in identifying the right funding scheme fitting to the scientific profile or project needs, in proposal writing as well as in budget calculation.

Yearly, K. Hübel is organizing together with V. Tegethoff from the Max Planck Institute for Radio Astronomy (Bonn) and the National Contact Point ERC a Workshop “Writing competitive ERC Starting and Consolidator Grant proposals“. This workshop is a special offer to junior scientists at Max Planck institutes in North Rhine-Westphalia and always well attended.

K. Hübel regularly gives a talk “Competitive Proposal Writing“ in order to qualify the researcher for writing high-quality, well-structured proposals.

Interview training

Especially applications for group leader funding often include interviews. Those are intensively practiced with participation of the research coordinator, the PR officer (see p. 104) and of course experienced scientific colleagues.

Project management

Support in project management was also given. This included assistance in setting scientific project reports as well as in financial reporting. Project reporting was done in close collaboration with the MPIE administration.

Career development

A special focus was set on the career development of young scientists. This included not only finding the right funding scheme that fits to their scientific profiles but also strategic advice for their career development. K. Hübel is supporting the researchers in doing a personal strengths & weakness profile to find the right direction. This includes also the option to use the respective skills of each researcher in diverse fields around science (e.g. research coordinator, consultant, founding a start-up).

Fig.1: *B. Gault (in front) received the Gottfried Wilhelm Leibniz Award 2020. On the picture he is seen with his team ready to celebrate the award.*

In general, support was given to junior scientists working at the MPIE as well as to prospective junior scientists planning to work at the MPIE. Together with the female scientist mentor C. Scheu (see p. 96) special support was provided to female senior postdoctoral researchers as well as young group leaders on their way to become a professor by giving focussed seminars on topics like scientific CV, competitive proposal writing or group leader funding.

Selected research funding (2019-2021)

B. Gault was awarded the Gottfried Wilhelm Leibniz Award 2020 of the German Research Foundation (DFG) which is the highest German research award.

Y. Ma received the Walter Benjamin Grant of the DFG. He was one of the first grantees awarded in frame of this new funding scheme.

C. Scheu got funding from the Federal Ministry for Economic Affairs and Energy: PAULL - Activation of ultra-long lifetimes of fuel cells.

S.-H. Zwaka won the DFG competition on international research marketing with her concept: Coffee with Max Planck: Research Opportunities at the MPIE.

Promotion of young scientists

K. Hübel

Research Coordination Office

We are highly committed to promoting young scientists at all career stages. The success of our intense coaching and training initiative is outlined in the following:

Scientific honours and apprenticeship awards

In 2020, B. Gault was awarded the Gottfried Wilhelm Leibniz Award of the German Research Foundation (DFG). In the same year, the MPIE was ranked first in Engineering Sciences in the Humboldt ranking. It was also ranked 3rd place over all sciences of non-university research institutions. Between 2019 and 2021 our young scientists got more than 50 prizes, awards and grants like the Walter Benjamin Grant from the DFG. The great success of our researchers is also due to the expert support in writing competitive proposals of our research coordinator K. Hübel (see p. 95). Also, our apprentices got honoured: from 2019-2021 they were awarded twice with the Apprentice Prize of the Max Planck Society (MPG) as well the Apprentice Prize of the Chamber of Commerce Düsseldorf. In addition, our young materials testers won the first place at the Innovationssemester competition of the Wissensregion Düsseldorf e.V.

Scientific output

The success of our intense coaching and training initiative is reflected by high scientific output: Between 2019-2021 junior scientists were author for 4 Nature papers, 23 Nature Communications, 4 Nature Materials and 6 Science Advances papers. Our young scientists are encouraged and financially supported to present their research results in international conferences, which is in addition supporting their networking activities and their recognition within the scientific community. During the Corona pandemic, our young scientists continued to present their research results in a variety of international virtual conferences and learned how to communicate and network in diverse online formats.

In the past 3 years, more than 20 of our scientists secured international professorship appointments at leading research institutes and universities like the Imperial College London/UK or the Karlsruhe Institute of Technology (KIT)/DE. We support our senior postdoctoral researchers as well as our group leaders in doing their next career steps. The directors are sharing their expertise on selection committees for becoming university professor by training the interview situation with the respective researcher. Our research coordinator, K. Hübel, is assisting our researchers in career planning. C. Scheu is the “MPIE Female Science Career Mentor”. Together with K. Hübel, J. Duarte (group leader), and S. Degner (gender equality officer, p. 99) she supports female senior postdoctoral researchers as well as young group leaders on their way to become a professor. Between 2019-2021 5 of our female scientists were selected to participate in the MPG Sign up! programme preparing them for leadership positions.

Career talks from our network

A variety of lecture series have been set up to support our young scientists in their career:

- Career talks by university professors who were MPIE scientists formerly
- Alumni talks by scientists in industry and academia organised by the imprs surmat
- „Be your own boss – how to found a start-up”: scientists who successfully started their own business share their experience

Teaching and supervision

Teaching expertise and supervision activities are important prerequisites for an academic career. We encourage our researchers to do both which is facilitated by our collaboration with the Ruhr-Universität Bochum and the RWTH Aachen University. To prepare our young scientists for lecturing, teaching rehearsals for postdocs were organized by H. Zhao to receive constructive feedback from their colleagues.

From researchers for researchers

We convey the importance on acting in concert and of efficient interdepartmental research to our junior scientists – a spirit which is fortunately taken over. Our PhD students organize MPIE lectures, where the group leaders and directors share their knowledge on different experimental and theoretical methods to the audience. Also, interdepartmental meetings are organized on a regular basis to further develop our interdepartmental research projects. In addition, the MPIE supports the PhD representatives in organizing diverse soft skill trainings.

Science communication

The institute’s PR officer Y. Ahmed Salem (see p. 104) is teaching our young scientists in communicating their science to different target audiences. Researchers get a PR coaching if they are invited to give an interview or if a TV-movie is made to present their research. In 2020, six researchers had the chance to explain their latest publications in professional films. Five of our young scientists won the communication award in frame of our internal video competition. They will explain our science to a non-scientific public in professional videos and act as science ambassadors. For this purpose they will receive a professional training on storytelling skills. All videos will also be published on our social media channels and serve as outreach activity for the scientists’ CV.

International Office

S.-H. Zwaka

Research Coordination Office

The International Office supports the MPIE's international researchers in their relocation to Düsseldorf and during their stay in Germany.

About 160 international researchers from more than 30 countries, mostly from outside the EU, conduct research at the MPIE. S.-H. Zwaka supports them - in her function as International Officer - from the moment they receive their job offer onwards.

To ensure a smooth relocation to Düsseldorf, the International Office assists with the visa process and keeps close contacts to landlords of apartments near the MPIE to help researchers find housing.

From the point of their arrival to the end of their stay, the International Office is their point of contact regarding all aspects of everyday life in Germany, including German immigration law, social insurance, taxation, housing and child care. Information is provided by personal consultation, via email as well as through the institute's intranet.

In addition to the personalized information, the International Office organizes events on topics relevant to international researchers' life in Germany. Events and informative sessions held in the past were for instance:

"Welcome Coffee +", a get-together to better integrate new international researchers.
"How to: Business trips", an informative session on applying for and filing expenses for business trips.
"Info session on pensions in Europe and VBL" held by speakers from "findyourpension.eu".
"International BBQ", the institute's interdepartmental summer party.

Since its start in 2018, the International Office has reached out to the Welcome Center of the Heinrich Heine University (HHU) Düsseldorf to allow for synergies in catering to the needs of international researchers in Düsseldorf. The MPIE's international researchers have since then been admitted to participate in informative sessions organized by the HHU's Welcome Center, such as:

"German Taxes", a session on German income taxes.
"German right of residence", a session of residence titles held by a representative of the immigration office Düsseldorf.

The International Office also coordinates the participation in German courses for the institute's international researchers.

The German courses are held at the HHU and the expenses are covered by the MPIE. In cases where courses cannot be taken at the HHU, there is the option to be partially refunded for courses taken at private language schools.

The MPIE's International Office is also part of the Max Planck Society's (MPG) International Office network and the Euraxess Germany network. The participation in those networks allows for quick sharing of information regarding visa, immigration and social insurance law in Germany. This has proven especially helpful during the Covid-Pandemic, when legislation regarding international travel changed quickly.

To ensure the smooth solution to complicated visa cases, S.-H. Zwaka is also in touch with representatives from the immigration office Düsseldorf.

To better accommodate the partners of international researchers, the International Office refers them to Düsseldorf's Expat Service Desk, where they can receive consultation on job search in Germany. The service is free of charge for qualified expats residing in Düsseldorf.

In 2019, S.-H. Zwaka was awarded the "International Research Marketing prize" funded by the German Federal Ministry of Education and Research (BMBF) for the concept "Coffee with Max Planck", a seminar series which aims at recruiting talented young researchers to the MPIE.

PhD Representatives

R. Dsouza[1], L. Frommeyer[2], A. Gedsun[2], M. C. Paulantonio[3], S. Pemma[2],
M. Poul[4], N. Samiseresht[3], E. Woods[3]

[1]CM, [2]SN, [3]MA, [4]GO

The PhD Representatives are a group of engaged PhD students across all departments at the MPIE. Our task is to advocate for the interests of PhD students at the institute and to build a strong network.

Fig. 1: *PhD student events and collaboration [1-3].*

The external PhD representative gets elected each year and represents the PhD students of the MPIE at the Max Planck Society. We are involved in the PhDnet, the platform for all PhD students across the Max Planck Society, and closely follow their activities. The participation in the PhDnet Survey and the follow-up discussions about the survey results with both, students and institute directors is an important collective involvement towards better working conditions at the institute. We see our role as an essential contribution to the PhD students, not only in their working life, but also to help them together with the International Office in settling when arriving from a new country/city, and in getting to know our institute's structure. We also organize social activities, such as board game nights and welcome events for new students. There, we inform newcomers about important things to know in our institute and all aspects of local life. Our online PhD portal provides the latest news around student life and relevant administrative help, e.g., enrollment at local universities and PhD defense procedures.

During the Corona pandemic, our social activities, meetings, and lectures moved to online platforms. The PhD Representatives organized an online prediction game for the Euro 2020 involving all employees at our institute this year. We are looking forward to a better situation where in-person meetings can be easily planned and re-engage in helping to organize our excursions, Christmas party, and barbecue events. Moreover, we are responsible every year for our Institute's merchandise, ordering and distributing hoodies and T-shirts with our logo for everyone interested in it.

We collaborate closely with the doctoral program of the institute IMPRS-SurMat. Some of their events, like regularly organized soft skill courses or excursions, e.g., the visit to Thyssen Krupp steel plant in 2019, are also open to non-SurMat PhD students.

The PhD Representatives have regular meetings with our institute board of directors, group leaders, and the works council where we discuss and address topics important to our colleagues. Improving working conditions has been one important issue in the last years. To this end, we also organized an online meeting for all PhD students with our administration department to explain different contract conditions. One of our main future focuses will continue to be the elimination of salary disparities that exist among PhD students.

References

1. N^2-PhD networks of Max Planck PhDnet, Helmholtz Juniors and Leibniz PhD Network.
2. PhDnet Max-Planck.
3. IMPRS-SurMat-International Max Planck Research School for Interface Controlled Materials for Energy Conversion.

Equal Opportunities/ Gender Equality

S. Degner

Administration

During the last three years the MPIE has consequently worked on the realization of its gender equality plan with the following main focus

1. reconcile the demands of family and career
2. raising awareness with regard on gender aspects
3. development of gender-responsive structures (e.g. recruitment-processes)

In autumn 2020, S. Degner was re-elected as Gender Equality Officer; a substitute could not yet be identified.

Here are the main measures - following our equal opportunities strategy - taken since 2019:

- parent-child-room, installed in spring 2020
- Gender Equality Office with fixed consultation hours once per week
- promotion of gender & diversity awareness at all levels (target group-oriented seminars and talks/ presentations)
- implementation of gender-equitable language: to be used in all documents/ statements; further actions taken for special target group by a programmed instruction
- accompanying female scientists on their academic career path/ coaching since 2019 (with C. Scheu)

Excellent basic research and the promotion of young talents are the pillars that shape the work at our institute. However female scientists often decide against a career in academic research despite their talent and excellent performance.

The promotion of women with regard to their professional development is therefore one of the main areas of our gender equality work. In addition, female scientists with their professional achievements and innovative ideas will be presented more visibly than before, both internally and externally, in order to be perceived by all parts of society as high achievers and high potentials and thus to be able to act as role models and impulse providers.

For this reason, our institute has worked on several approaches to improve the existing situation.

C. Scheu, leader of the independent research group “Nanoanalytics and Interfaces“ is mentor for senior female scientists. She is also member in our working group “female scientists at MPIE”, together with K. Hübel, head of the Research Coordination Office, J. Duarte, leader of the group “Hydrogen Mechanics & Interfaces”, and S. Degner, Gender Equality Officer. The group meets once per month and focusses on improving the situation for female scientists and to support them actively, e.g. by training of interviews for becoming a group leader or professor. First initiatives were:

A talk on “Obstacles in the scientific career of female scientists - reports of experience” by a female professor (S. Korte-Kerzel) from RTWH Aachen University -followed by intensive discussion- in February 2020. Target audience were all group leaders (female and male) at the MPIE.

In January 2021, we realized a 2-day career seminar about communication and leadership, which was very fruitful and in consequence we established the “female scientist lunch”. The group of mentees meets regularly to address and discuss topics of interest for them, e.g. “Pregnancy during my scientific career”. Furthermore, we had topics like “Drafting a good scientific CV”, “Writing a competitive proposal” or “Group Leader funding”.

In addition, awareness enhancing online events on the following topics were held in 2020:

1. gender: “What does gender have to do with physics?” talk with following discussion by T. Brage, Physics Department, University of Lund /Sweden
2. diversity: “No sexuality please, we are scientists” talk by B. Britton, materialist scientist from Imperial College, London /GB followed by a discussion.

Both formats had a huge audience (about 80-90 participants) with an extended discussion afterwards, what has shown the demand for these formats at our Institute.
Further action will follow in autumn 2021 aiming for ethnicity aspects and on (in-)visibility of female scientists (papers, publications etc.)

Family service

S. Degner[1], *S.-H. Zwaka*[2]

[1]Administration, [2]Research Coordination Office

Copyright: pme Familienservice

With the aim of supporting our employees in maintaining a good balance between family and career, the MPIE offers and further develops several measures to better reconcile work and family life.

One pillar is the "pme-Familienservice", also contracted by our mother organization, the "Max Planck Society for the advancement of Science" (MPG).

The "pme Familienservice" offers information and support on different aspects of childcare, such as: Finding childcare, emergency child care in case of illness of a child and au-pairs. It also offers a holiday programme for the employee's children to better bridge the time that child care facilities are closed during holidays. Furthermore, it offers seminars on parenting and juggling career & care work. The other subject of pme is covering elder care aspects. Similarly, the "pme Familienservice" helps to find nursing staff for elder care or places in a care-home. The costs for counselling and mediation services are borne by the institute.

Copyright: pme Familienservice

To react to the corona crisis, the institute expanded the family service contract on an online training and education programme.

The second pillar of our support measures is the childcare offer of the MPIE. Since 2017, the institute reserves between two to four child care places per year in two child care facilities near the institute for children aged one to three years. This is especially useful for international researchers moving to Germany with their families and for those who are unfamiliar with the German childcare system. To complement this, the institute also informs about the German child care system and consults researchers on how to find childcare in Düsseldorf, apply for public child benefit as well as public parental allowance after the birth of a child.

Young scientists can apply for the MPG childcare subsidy for infants. PhD students and postdocs can submit their applications to the MPIE to receive a subsidy of the costs for crèches, childminders or babysitters who provide an official bill for their services. The subsidy funds up to fifty percent of the costs - maximum 400 Euros per month. To be eligible, both parents need to work a minimum of fifty percent and toddlers should be aged between three and twelve months.

Corporate health management

C. Rogge

Administration

Excellent research needs excellent scientists. Excellent scientists are highly qualified experts in their field. However, they can only utilize their potential when they are healthy, motivated and happy in their job. Therefore, the people's health at the MPIE is a crucial factor for the scientific success of the institute.

The responsibility of the corporate health management (CHM) at the MPIE is to evaluate continuously the personal situation of the employees and their working environment as well as their wishes and requirements. The CHM-Team aims to preserve and raise awareness for this important resource as well as harmonizing personal well-being with working conditions. Several measures have been taken:

Since 2018 MPIE, Health Days are taking place to ensure that all colleagues are informed about the activities of the CHM and to raise the awareness for the importance of healthy working conditions. Methods of health analysis (posture diagnostics, back check analysis, body stability test) and training for health improvements were offered. Because of the Corona pandemic, the Health Days could not take place in 2020 and 2021.

Fig. 1: *Employees are taking part at a gymnastic unit to implement easy exercises into their daily life. Gymnastic units are now offered twice a week at the MPIE as a so-called animated break during work.*

Corporate health management needs data

As targeted suggestions need data the CHM carried out a survey on Psychological Stress at Work. There was a good participation rate of 54 % out of 270 colleagues.
The overall results showed that the working conditions at MPIE are predominantly rated as above average.

The following categories were emphasized

- Attachment to the workplace/ job satisfaction
- Influence on the work
- Development opportunities
- Leadership quality
- Trust/fairness/appreciation

The measures in the context of the Corona pandemic were also rated very positively. There are areas that should be investigated more deeply and that could be improved:

- Disengagement (doing work-related things outside of work hours, being available at work during free time)
- Work-privacy conflicts
- Burnout symptoms/ inability to switch off

However, all three identified areas only slightly deviated from the average.

Corporate health management initiatives

Due to the study several measures are planned or have been already implemented e.g. a vaccination was offered. The vaccination campaign was organized and accompanied by K. Kudlich (Company Safety Officer).

The appointment of "health ambassadors" is planned. The ambassadors shell keep contact and inform the members of the different departments concerning corporate health measures.

Based on the finding that physical fitness is an important factor for the personal well-being, the following steps were initiated:

- Motivational speeches with exercises "My moving workplace" and "Ergonomics at workplace" by D. Nyarko.
- Impulse lecture "My back and me".
- Workshops e.g. concerning "Mental and physical health at workplace" and "Healthy Leadership".
- Prevention measures for stress relaxation in form of exercises like Qigong/Daoyin, Tai Chi Chuan and back training and relaxation.

The Fürstenberg Institute offers free of charge psychological support in form of anonymous ad hoc one-on-one coachings to MPIE co-workers.

In addition, the CHM supports the integration into employment of temporarily sick colleagues.

Alumni networking

B. Kohlhaas

GO

In over 100 years of MPIE tradition, thousands of employees have worked and researched in various areas and departments, thus contributing to the institute's success.

For many scientists, the Max-Planck-Institut für Eisenforschung (MPIE) was an important milestone in their career where they established scientific contacts and built friendships. We take a great interest in the career of our alumni and thus have created the MPIE alumni network.

Alumni - who are they?

Everyone who has worked at the Max-Planck-Institut für Eisenforschung can join the alumni network.

For us at the MPIE, alumni encompass all former employees: experienced and junior scientists, colleagues from the scientific support sector, "retirees" who continue to be active in the field as well as former guest scientists – in short: everybody who wants to stay in contact even after their time at the Institute.

Alumni network - what is it?

In order to promote close contact and lively exchange among former institute members and active staff, the alumni network of our institute was founded in 2003 with currently more than 1000 members from over 30 countries. Membership in the alumni network is free of charge.

Alumni membership – what are the benefits?

Our alumni represent an integral part of our network. They are informed about MPIE-related news, events and activities via e-mail. Regular notifications, e.g. via the MPIE newsletter or through social networks (LinkedIn, Twitter), will keep them up to date on topics at the MPIE as well.

The alumni network offers opportunities for joint events, publications and projects beyond the alumni´s time of employment at the MPIE.

Other options for cooperation include guest visits, mentoring or scientific collaboration.

Fig. 1: *Networking at the 100 years anniversary (K. Velmans, MPIE GmbH).*

Alumni networking – what are the aims?

Our aim is to further expand the existing network in order to continually strengthen alumni relations both with the MPIE as well as among our alumni across fields of work and generations, across national borders.

A network of this kind enables all of us to initiate and develop cooperations; it brings about the involvement of alumni in research and, last but not least, contributes to career promotion.

Keep in touch

In addition to registration in our alumni database, alumni are offered the following opportunities to network with other MPIE alumni and to get actively involved:

- MAX Intranet (MPIE)
- by e-mail (alumni@mpie.de)
- Alumni news at https://alumni.mpie.de
- Social networks (LinkedIn, Twitter, XING)
- Max Planck Alumni Association e.V. (MPG)

Sustainability in the work life: Team Green and the we4bee project

B. Breitbach[1], *E. Gattermann*[2], *H. Springer*[3]

[1]SN, [2]RCO, [3]MA, RWTH Aachen University

Every day, scientific progress at the MPIE advances our understanding of materials aiming for a sustainable future of our society. Equally important, however, is a sustainable and ecological working environment. That motivated us to launch our participation in the we4bee project in 2019. The Team Green started its activities in 2020.

The we4bee project

The decline in the insect population as part of a biodiversity crisis is starting to become noticeable in Germany. Insects are not only an important part of the food chain as a protein source for various animal species, but they also occupy pivotal functions in the ecosystem, such as the pollinating of plants. It follows that an adequate supply of food for mankind is directly dependent on a healthy insect population.

Fig. 1: *Bee house at the roof of hall 8. Cameras and high-tech sensors explore the life of the bees.*

An important first step to counteract this problem is to raise general awareness of the topic of insects and biodiversity. Honeybees have a very positive image, mainly due to their high pollination capacity, but also for their production of tasty honey and useful wax. They therefore represent ideal ambassadors for the insects as a whole.

This approach is used by the we4bee project, initiated by the emeritus biologist J. Tautz from the Julius Maximilians University of Würzburg. With the help of various teaching, educational and research institutions, this project aims to bring knowledge about honeybees and their importance for our ecosystem to as many people as possible. The project provides beehives equipped with modern sensors to be set up at various locations. With the help of the sensors, the most important data of the bee colonies, as well as weather and environmental data are collected. The members of the facilities take care of the individual bee colonies on site. The data is recorded centrally online by the University of Würzburg and then used to clarify various scientific questions.

The MPIE applied successfully as one of the first hundred locations at the start of the project in 2019. In July 2019, B. Breitbach, a technician at the institute and hobby beekeeper for many years, settled a colony of bees on the institute's premises, initially in a conventional beehive to allow them to settle in. A part of the roof of Hall 8 was chosen as the installation site, as the bees are not expected to disrupt the institute's operations and vice versa. In December 2019, the beehive equipped with scientific measurement technology was delivered. Since the move of the bee colony into its new scientific home in May 2020, the MPIE is contributing its data to the we4bee project.

The Team Green

The MPIE's sustainability group set up in 2020 under the name Team Green, and is part of the MPG's sustainability network. The goal of the group is to help the working environment at the institute become more sustainable and environmentally friendly while making the work itself more pleasant as well as supporting the local ecosystem. The initiative is supported by the directors of the MPIE.

The first successfully implemented project of Team Green was the introduction of consistent waste separation and the proper disposal of empty toner cartridges. For this purpose, yellow garbage cans for packaging waste were introduced, a collection point for empty toner cartridges that is accessible to all employees was set up, and corresponding informational material was published on the institute's intranet and on notice boards.

In 2021, part of the institute's grounds will be re-vegetated with insect-friendly flowering plants. This will benefit the insect population in general and the flying members of the we4bee project in particular.

Other issues the group plans to address are mobility (business trips and infrastructure for bicycles) and energy consumption.

Public outreach

Y. Ahmed Salem

Research Coordination Office

Communication is getting faster, interconnected and reaches more and diverse people through nearly overall available technology. The same applies to corporate communication, which has to be at the pulse of time. The MPIE's press and public relations has experienced a vast change in the reporting period 2019-2021. It reaches out to a broader audience through newly set up social media channels and responds faster and more precise to evolving topics in research and society through agenda surfing. How does this happen?

Fig. 2: *Behind the scenes – A. Garzon is explaining her research about catalysts. The film is available on our YouTube channel.*

Website and social media

The MPIE's website has been relaunched in 2020 to a responsive design, which enables users to surf through the website with any device without experiencing loss in style or information. Moreover, the relaunch comprised an update of the overall style to a more modern appearance in accordance with the corporate design of the Max Planck Society. The website now also comprises so-called tag lists, which sum up all projects and news about our hot topics sustainability (with the subtopic hydrogen), digitalization, microstructure-property relations, innovative materials, materials under harsh environments and advanced method development (https://www.mpie.de/4197842/key-topics). These lists are automatically updated and serve e.g. journalists as easily accessible sources of information.

Fig. 1: *Linkedin post design for the campaign "Women at MPIE".*

The MPIE now also hosts several social media channels to reach early career researchers and students. The aim is to recruit new talents and increase the institute's publicity. A Linkedin channel was set up in March 2020 with 3813 followers by December 2021, mainly from industry and academia. The twitter channel, which was set up in April 2021, currently has 481 followers and our new YouTube channel (since July 2020) has 639 followers. The Linkedin and Twitter channels do not only show adapted content from the MPIE's website, but also highlight content which is not displayed on the website as news or press releases. This content comprises e.g. selected publications, job openings, and a new social media campaign "Women at MPIE", which presents every two weeks a MPIE female scientist and her path into research. The MPIE YouTube channel presents videos made by the PR of the institute, by the researchers themselves or professionally made videos. This has been enforced by a very successful cooperation with an external agency funded through the Max Planck Society. The MPIE scientists are selected by the PR officer and the directors and get their own video interview highlighting one of their recent publications. In 2020, a new recruiting film was made highlighting some of our hottopics in research and why it is worth working at the MPIE. Moreover, in 2021 the PR officer organized an inhouse science video competition. The five winners R. Aymerich Armengol, R. Changizi, A. El-Zoka, R. Hosseinabadi, and E. Ibrahim Saad will get a storytelling and camera training in January 2021 and will each be filmed presenting one of the key research topics of the MPIE. They will act as science ambassadors for the MPIE and can benefit from the films and training for their CV and communication skills.

Corporate design

Design trends keep on changing and so does our corporate design. We have a new design since the end of 2020 with new templates for posters, presentations etc. and with a new logo. The design is based on the corporate design of the Max Planck Society (MPG) to be easily recognized as a Max Planck institute and support a strong brand communication. The MPIE's logo, which consisted of the Minerva (logo of the MPG) and the symbol for iron metallurgy, was adapted to the new funding scheme of

Fig. 3: *Development of the MPIE logo: left) from the beginnings till 2017 middle) from 2017 till 2020 to celebrate the 100 years anniversary and right) in accordance to the new financial situation of the institute and the new corporate design.*

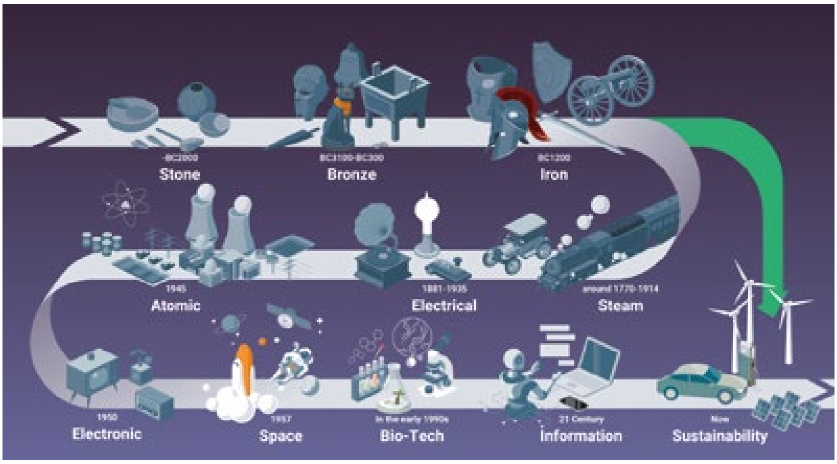

Fig. 4: *Developments in metallurgy are now more needed than ever. The picture is an example of the illustrations made by T. You, a graphic design student hosted by the PR.*

the MPIE and comprises now only the Minerva (Fig. 3). The PR now also hosts a student graphic designer, T. You, who helps in creating new eye-catching illustrations (Fig. 4), which are especially helpful in social media communication. Moreover, the website design goes hand in hand with the corporate design thus enforcing a high recognition value.

Press relations

The press relations of the MPIE consist of press releases, short news, articles and other printed and online publications, which are written or supervised by the PR Officer. Press releases are send out to journalists, published on the MPIE website and on the idw, an online service offering latest news to a wider range of interested journalists and people from industry and academia. They are also summed up on our new social media channels. A special publication is the MPG yearbook, where all Max Planck institutes contribute with texts about their research. Only 15 out of more than 80 articles are selected by the MPG and printed in a special highlight book, which is distributed to politicians. The MPIE's texts were selected twice in the reporting period for this special highlight book with texts about additive manufacturing and sustainable alloys. The MPIE's research is getting more attention, also from TV and radio channels. We had 2 interviews for TV broadcast, one about our we4bee project (September 2020, see p. 103) and one about green steel (July 2021); and one radio interview about iron aluminides (June 2021). Besides the articles in newspapers, two publications are worth mentioning: an article about corrosion published in the journal GEO (October 2019) - a journal that presents detailed documentaries about diverse topics. And an article about additive manufacturing published in the MPG journal TechMax – a journal for teachers and pupils (November 2019).

Internal communication

The internal communication was characterized by the crisis communication due to the Covid pandemic and a relaunch of the intranet from a self-maintained platform to the platform provided by the MPG. The Covid pandemic made it necessary to provide up to date information on all regulations published by the German government and adapted in the institute like home office rules, travel regulations, vaccine possibilities etc. These regulations were distributed via email to the MPIE staff and collected on the intranet. The PR also summed up all regulations in a FAQ and supported the MPIE board in answering all evolving questions, especially during the first months of the pandemic.

The intranet relaunch took place in August 2021. The idea was to have an easily accessible platform for all MPIE members to get information on internal matters and the services and information provided by the MPG. The new intranet is now the gateway to the whole Max Planck world. Main MPIE services and contact persons are presented and the respective persons in charge of a certain topic can maintain their intranet pages by themselves. This was possible through trainings for all persons who present information on the intranet and guarantees that the available information is always up to date and does not depend on the availability of the PR officer.

Events and special guests

The PR organized several live events before the beginning of the Corona pandemic. We had three KopfSalat events, which is a series taking place twice a year at the MPIE and inviting speakers from all fields of research to give gener-

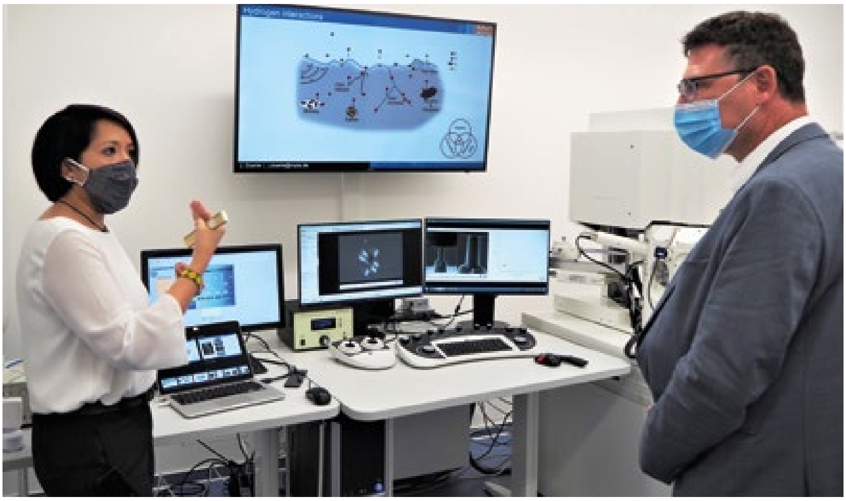

Fig. 5: *M.J. Duarte Correa is explaining her research on hydrogen resistant coatings to S. Kaufmann, delegate of the Federal Ministry for Education and Research in September 2020.*

ally understandable talks to a broad public with the aim to foster the popularity of the institute in and around Düsseldorf. Nobel Prize Winner S. Hell (Max Planck Institute for Biophysical Chemistry, Göttingen) gave a KopfSalat talk in April 2019 about advanced microscopy. S. Zaefferer (MPIE) gave a talk about crystals in metals in September 2019 and T. Bertram (Technical University Dortmund) about self-driving cars in February 2020. The talk of S. Zaefferer took place in frame of the Düsseldorf Researchers' Night, where the MPIE also presented its work on additive manufacturing and on measuring material properties. Moreover, the doctoral researcher A. Dutta participated at a science slam and won the second prize. The Girls Day was organized for March 2020, but did not take place due to the Covid pandemic. However, the institute was able to participate for the first time at the Pint of Science event in May 2021, which took place online with the MPIE researchers B. Bellon, Ü. Güder and V. Devulapalli. In cooperation with the MPG, D. Raabe was also able to give two online talks on sustainable metallurgy in November 2020 and March 2021 targeting a broad public.

The PR also organized several lab tours for students, scientists and politicians like a tour for a student group from South Dakota, USA, and for delegates of the Korean Steel Association, both in January 2019. Furthermore, several politicians concerned with sustainability and hydrogen economy were invited to the MPIE, like S. Kaufmann, delegate of the Federal Ministry for Education and Research for green hydrogen.

A special project: historical review

The MPIE celebrated its 100 years anniversary in 2017. In preparation for this anniversary, an agency specialized on history reviews was tasked to research about the institute's history. During this research, the agency discovered unknown insights into the years of Nazi occupation. These insights were the reason to begin a new project about the events during this critical phase of the institute. In coordination with K. de Weldige and Y. Ahmed Salem, the agency studied diverse archives and finally summed up all findings in a publicly available wiki and a detailed manuscript, available on demand. The wiki offers detailed information on persons, historic events and research of that era. The wiki is available on the MPIE's website: https://www.mpie.de/4026021/geschichte. It is also planned to organize a KopfSalat talk with the historians to inform people transparently about the past happenings and thus educating future generations.

Part II.

The departments

Department of Computational Materials Design (CM)

J. Neugebauer

Scientific Objective and Department Structure

The research activities of the department Computational Materials Design rest on three pillars: (i) **development** and implementation **of advanced *ab initio* guided methodologies and algorithms**, (ii) **application of these tools** to address **fundamental materials science questions**, and (iii) utilization of these insights to **explore and identify novel strategies in materials design** and synthesis. Based on these pillars fundamental mechanisms controlling the microstructure, the properties and the synthesis of a wide variety of materials, ranging from metallic alloys over semiconductors for optoelectronic and high-power electronics to electrochemical systems, could be – often in close collaboration with our experimental partners at the MPIE – successfully studied.

In the reporting period several major successes have been achieved: Our **recent developments in advanced numerical/physical algorithms, machine learning and automated workflows** opened the opportunity to study structurally and/or chemically complex materials systems that were until recently out of reach for *ab initio* based simulation methods. Examples are **compositionally complex materials** consisting of four or more chemical species [1-14; see p. 210, p. 194, p. 208], **electrochemical interfaces** as relevant for corrosion or energy conversion [15-18; see p. 224, p. 69, p. 198], the structural, mechanical and chemical interplay at crystal defects resulting in **defect phases** [19-24; see p. 192, p. 196, p. 206] and the impact on **mechanical properties** [25-27; see p. 194] and the mechanisms controlling the **resolution and interpretation of advanced experimental techniques** such as, e.g., atom probe tomography (APT) [28-31].

Many of these applications became only possible by systematically extending our in-house developed **materials science integrated development platform pyiron** [www.pyiron.org; see p. 72; 32-36]. In the last three years pyiron has evolved as a highly efficient tool that provides and combines all steps occurring in the development and application of our advanced simulation techniques. The expertise gained by developing pyiron as well as its rapid prototyping, data handling and high performance computing capabilities brought the department into an ideal position to **successfully initiate and join large scale digitalization projects in industry and academia**. Examples are the **National Research Data Infrastructure (NFDI)** initiative (see p. 81), which aims to establish a unified research data infrastructure for universities and academic research organizations, or the **Platform MaterialDigital** (see p. 80), which wants to provide such an infrastructure for industry. It also helped to establish large scientific networks such as the **Collaborative Research Centre on defect phase diagrams** (see p. 76) by providing the necessary data and workflow infrastructure. Many of the scientists working in these projects are highly active in the further development of pyiron. They formed a board of core-developers that together with external developers outside MPIE introduces, supervises and directs the implementation of new features, simulation tools and ontologies.

This newly developed **expertise in big data analytics, machine learning and software engineering** made the department attractive for scientists interested in combining this expertise with materials science concepts and changed partly its scope and composition. It also allowed the department to **successfully extend the powerful concepts developed in previous years**. Examples are the extension on the fully *ab initio* computation of key thermodynamic quantities such as free energies **from unaries to compositionally complex alloys** [19-20, 27, 37-45], the **transition of labour intensive computations into a fully automated approach** to determine precise melting temperatures [36], a correction schema to enable accurate *ab initio* calculations of the technologically highly relevant class of semiconductors exhibiting spontaneous polarization [46], or to model the impact of the extreme electric fields occurring in atom probe tomography [28, 47].

Utilizing machine learning concepts together with the above simulation techniques also opened a new class of **joint activities with the other departments to boost experimental resolution and interpretation**. For example, these techniques have been successfully used to achieve **chemical resolution for field ion microscopy** (FIM) measurements (see p. 220), to **reveal solute-vacancy interaction in APT** [29] or to design a **fully automated segmentation algorithm** for atomic resolution microscopy [30].

The rapid prototyping capabilities, the interactivity of pyiron, and the ease to create automated workflows also enabled the department to **develop and implement new group activities to handle the restrictions imposed by social distancing measures** due to the COVID-19 pandemic. The traditional department retreat was replaced in 2020 by a three-day hackathon, where small groups consisting of 3-6 scientists were identifying challenging materials science simulation topics and were jointly developing, coding, and testing algorithms and tools in interactive sessions. This first hackathon was so successful, that many of the participants proposed to make this a regular event. As a result, 3-5 groups with changing participants organize weekly two-hour **hackathons** on various topics, where members of the department **develop, program and discuss new simulation tools and algorithms** (see p. 216). Also, based on the success and the grass-root organization of these meetings a **Journal club** has been established, in which key ideas of selected publications are not only discussed, but actually implemented and tested. The highly interactive character of these meetings, enabled e.g. by Jupyter notebooks and pyiron, helped us tremendously in **integrating new members of the department** when home-office was enforced.

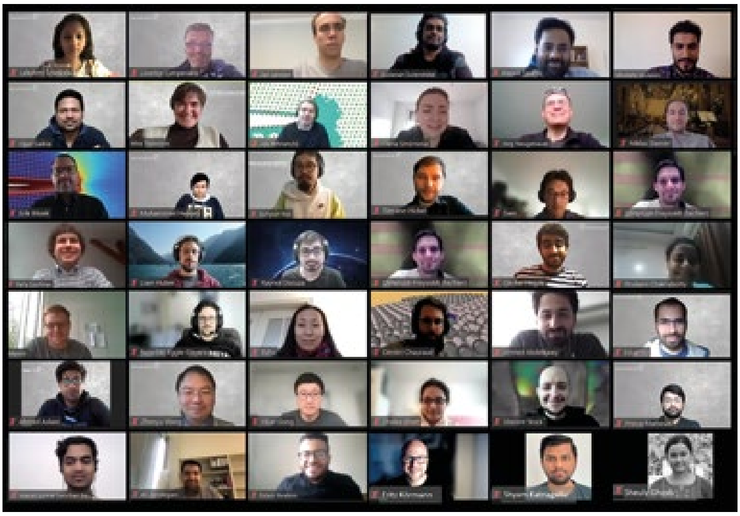

Fig. 1: *The CM department (2021)*

Many of the breakthroughs achieved in method development [15-17, 46-60], application [52, 61-71] or design [72-83] became only possible by **combining the individual expertise developed by the groups** in the department. Examples are the development of algorithms that combine spin-constraints and highly efficient atomic relaxation techniques (**Freysoldt group**) with high-performance statistical *ab initio* thermodynamics sampling approaches (**Grabowski and Hickel groups**). These algorithms and techniques are implemented in pyiron (**Hickel and Huber groups**) and applied to complex materials such as high-entropy alloys (**Körmann group**; see p. 55) or defects/surfaces and their phases (**Hickel, Huber, Lymperakis, and Todorova groups**). Also, combining the individual group expertise resulted in powerful new concepts and simulation tools, allowing to **extend available DFT** (density functional theory) **codes such that they describe a new level of physics and phenomena**. Examples are the development of a **thermopotentiostat** to realistically model the impact of extreme electric fields present at electrochemical interfaces (Wippermann (GO), Freysoldt and Todorova groups), a new approach to **boost size convergence for materials exhibiting spontaneous polarization** or piezoelectricity (Todorova and Lymperakis groups) or to **describe atomistic and electronic structure phenomena occurring at cutting edge experiments and that impact their resolution** (Freysoldt in collaboration with Gault (MA) and Liebscher (SN) groups).

The high international visibility of these developments and studies opened **prestigious job opportunities** for several members of the department. The head of the "*Ab initio* Thermodynamics group" and ERC Starting Grant Awardee (see p. 51) **B. Grabowski**, accepted a professorship position at the University of Stuttgart in 2019. **L. Lymperakis**, who is heading the group "Growth Modelling" received a professorship position at the University of Crete. **T. Hickel**, head of the "Computational Phase Studies" group, accepted a position as division head at the Federal Institute for Materials Research and Testing, BAM. To strengthen the collaborations and links between BAM and MPIE, with a particular focus on digitalization topics and large scale networking projects (see p. 80 & 81), he holds now a 50% position at both MPIE and BAM. **S.-H. Yoo** successfully applied for an Energy for Future (E4F) Postdoctoral fellowship – an initiative of the Horizon 2020 MSCA-COFUND Programme – to go to Imperial College London. **J. Janssen**, the lead developer of pyiron, graduated with distinction (summa cum laude) and started a PostDoc position at Los Alamos National Lab.
In the last period **two new groups have been established**: **L. Huber started in 2019 a group on the "Thermodynamics and Kinetics of Defects"** (see p. 41). In Fall 2021**, E. Bitzek**, formerly professor at the University of Erlangen-Nürnberg, **established a new W2 group on "Microstructure & Mechanics"** (see p. 37). His expertise e.g. in large scale atomistic simulations, fracture mechanics and mesoscale approaches ideally complements the electronic structure and atomistic expertise already existing in the department and opens exciting new links to the other MPIE departments as well as to external collaboration partners.

To foster collaborations with leading experts worldwide, members of the department actively engaged in **initiating and organizing international workshops and symposia**. Due to the Corona restrictions many of these were online workshops: Examples are two hands-on workshops utilizing pyiron. In one workshop with more than 250 participants it was shown how atomistic free energy calculations can be combined with CALPHAD concepts to obtain phase diagrams. Another hands-on workshop with ~100 participants was used to demonstrate all steps necessary to generate modern machine-learning potentials. Also, our successful biannual workshop series on "*Ab initio* Description of Iron and Steel" (ADIS; T. Hickel) took place in November 2020. C. Freysoldt was co-organizer of the BiGmax summer school, a MPG funded initiative to boost machine learning concepts in materials science. Furthermore, several symposia at international conferences like

Fig. 2: *The last in-person retreat of the CM department took place in October 2019 in Domburg (NL) with participation of colleagues from ICAMS and the TU Delft.*

the DPG (J. Neugebauer, F. Körmann, M. Todorova) and MSE (L. Huber) were organised. M. Todorova organized in Spring 2021 a virtual retreat together with partner groups at ICAMS (Ruhr-Universität Bochum) and Delft university consisting of presentations and interactive hands-on sessions. (see p. 247).

The high quality and visibility of their research enabled members of the department to receive **highly competitive and prestigious research grants**. **Z. Wang** received a FP-RESOMUS Postdoctoral Fellowship. **M. Todorova** was awarded with the Boost! Programme of the MPG that provides substantial funding and resources to propel the carrier of MPG group leaders. **S. K. Bavajigari** received a Humboldt Fellowship in Fall 2021.

Due to the expertise of the department in developing tools for big data analytics and advanced data storage with focus on computational materials science members of the department became highly active in **national and international community activities related to digitalization in materials science**. **T.Hickel** and **E. Bitzek** are heading the **NFDI-MatWerk Task Areas "Workflows and Software Development" and "Materials Data Infrastructure"**, respectively. **T. Hickel** and **J. Neugebauer** are Members of the **Platform MaterialDigital Steering Committee**. J. Neugebauer also served as an elected member of the DFG review board (Fachkollegiat) Materials science (until Summer 2020) and was elected into the supervisory board of the Karlsruhe Institute of Technology (KIT). Together with G. Eggeler (Ruhr-Universität Bochum) he is also **the speaker of the new Max Planck Research School on Sustainable Metallurgy (SusMet) starting in Jan. 2022** (see p. 74).

In the following, a brief overview of the activities and research highlights of the groups in the department is given.

Research groups

Computational Phase Studies (T. Hickel)

The group "Computational Phase Studies" is devoted to the physics of (meta)stable thermodynamic phases in metals as well as transitions between them. Its major vision is an ab initio based prediction of thermodynamic bulk and defect phase diagrams, being directly related to many technologically relevant properties and processes in metals. Over the last three years, the group made again substantial progress in developing methods and applying them to advanced material systems. This applies in particular to the following fields of research:

- The desire to push ab initio thermodynamics to its limits, to resolve, for example, the coupling of thermodynamic, chemical and mechanical degrees of freedom, the stabilization of structural modulations, the life time of quasiparticles, or hitherto unclear features in experimental data.
- The prediction of phase stabilities next to extended defects, such as grain boundaries, stacking faults and interfaces.
- The chemo-mechanical coupling during precipitate growth in structural materials, such as steels and Al alloys.
- The application of the developed thermodynamic methods to novel functional material systems.
- The development of generalized workflow solutions that make the simulations in the above-mentioned fields transparent and reproducible.

The research in the group is more and more characterized by digitalization strategies within the department and well beyond. The main motivation for the group is to convey the innovative methodological developments from one project to the other and to provide a flexible environment that allows a quick adaptation to new ideas and concepts. The integrated development environment (IDE) pyiron is ideally suited for this purpose. Its philosophy and success have already been outlined before (see

p. 72). Particularly relevant in the context of the group is the deployment for the complex simulation protocols required for ab initio thermodynamics.

One of the fundamental concepts of pyiron is to consider data and workflows as a unity. In this way the FAIR (findable, accessible, interoperable, reusable) data policies, which are pushed forward by national and international consortia, are automatically fulfilled. The philosophy of pyiron is, therefore, a cornerstone of large-scale national initiatives like Platform MaterialDigital (see p. 80) and NFDI MatWerk (see p. 81).

To ensure the sustainability of the digitalization activities the group head (T. Hickel) received the offer to start a second employment at the BAM Federal Institute of Materials Science and Testing. His intention is to establish another centre for the development of workflows in materials science at a place that has the strategic mission of the government to ensure standards, quality, and safety in materials science. At the same time, BAM offers the opportunity to spread the developed digitalization concepts within one institution to a large number of stakeholders from different disciplines in materials science, including multiscale simulations and experiment, but also the interface to chemistry and engineering. BAM has the potential to give Platform MaterialDigital, NFDI-MatWerk and the workflow solutions therein a long-term perspective and operation centre, well beyond the funding period of these initiatives. In order to build up these new scientific and strategic developments at BAM, T. Hickel had to reduce his employment and time budget at MPIE to 50%, starting from 1 August 2021. Altogether, a substantial part of the group resources in the last three years has been invested in these digitalization activities.

In the field of ab initio thermodynamics, the previous achievements in handling all entropy contributions to the Gibbs free energy with highest-possible accuracy have been used to investigate the coupling of different degrees of freedom. One highlight was the prediction of phonon lifetimes, resulting from the interaction of phonons with other phonons [48], with chemical disorder [60], and with magnetic disorder [37] (A. Glensk, B. Dutta). These studies emphasize the dominance of local effects such as the anharmonicity of local pair interactions [48] as well as fluctuations of the local chemical environment [60]. The consideration of local magnetic fluctuations has revealed that the assumptions of a spin-space averaging approach are for realistic materials and time scales sufficient, to describe their phonon spectrum [37] (B. Dutta).

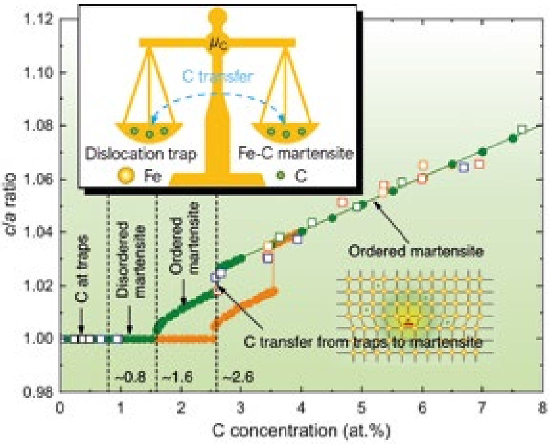

Fig. 3: *Comparison of experimental (open squares) and theoretically computed (filled circles) c/a ratio of Fe-C alloys as a function of C concentration at room temperature. Calculations with (green circles) and without (orange circles) considering anharmonicity have been compared. Figure adapted from [26].*

Our approaches in ab initio thermodynamics have strengthened the ab initio guided calculation of phase diagrams. As part of the efforts towards a third generation of Calphad databases, novel concepts for a parametrization of the solid phase beyond the melting point have been investigated for Al alloys [38] (A. Glensk). Furthermore, an assessment of magnetic models has been performed by a comparison with ab initio based Monte-Carlo simulations for hard-magnetic materials [72] (H. Sözen) and the applicability of empirical rules has been tested such as Neumann-Kopp in a temperature regime where anharmonic vibrations are relevant [39] (A. Gupta). Recently, thermodynamic approaches have been applied to predict the stability of chemically complex carbides (L. Sreekala). In addition, the thermodynamics of binary Fe-Mn alloys has been extended to machine learning approaches to handle the complexity of magnetic interactions in metallic alloys [62] (O. Waseda).

The thermodynamics of defects is a third major topic of the group that substantially gained importance after the establishment of the CRC1394 “Structural and chemical atomic complexity” (see p. 76). The extension of the concept of bulk phase diagrams to defect phase diagrams is one of the major visions of these activities [19]. Substantial breakthroughs in the development of these concepts have been achievement in the context of stacking faults in an off-stoichiometric Fe_2Nb Laves phase [20] (A. Zendegani). We have transferred the established convex hull representation to a phase diagram as a function of chemical potentials and have included the coherency strain as an additional degree of freedom (see p. 192). These concepts are currently extended to other material systems such as Mg alloys (A. Tehranchi, P. Mathews) and Al alloys (P. Chakraborty, A. Aslam).

The thermodynamics of defects in magnetic materials is particularly challenging due the different time scales of magnetic fluctuations and atomic relaxations. Taking this properly into account, has a substantial impact on the atomic relaxation next to vacancies and therefore the vacancy formation and migration energies [40]. The decisive role of planar defects has also been investigated in magnetic materials, where the presence of twin boundaries has been found to substantially reduce the size of magnetic domains in MnAl Heusler alloys [63] (O. Hegde).

Several investigations have again been devoted to mechanisms in structural materials. One of the outstanding findings, is related to the competition between interstitial ordering in Fe-C alloys and the segregation to extended defects such as dislocations (Fig. 3). We have predicted an effective decrease of this segregation for high interstitial concentrations, which was confirmed by atom probe tomography [26]. Other works are related to the kinetics in these materials as, for example, the multicomponent interdiffusion in Ni-Al alloys [60]. The research on hydrogen embrittlement of structural materials was boosted by an European RFCS grant on the "Hydrogen interaction with retained austenite under static and cyclic loading conditions" that is coordinated by T. Hickel. One outstanding finding within this project is the change of phase stabilities due to the presence of hydrogen (A. Tehranchi). Similarly, the interplay of hydrogen with carbides in steels has been investigated (L. Sreekala, Fig. 4). A fruitful collaboration with the group of B. Gault within the ERC project SHINE (see p. 50) has led to amazing insights into the competition of precipitates and grain boundaries in the hydrogen embrittlement of Al alloys (P. Chakraborty).

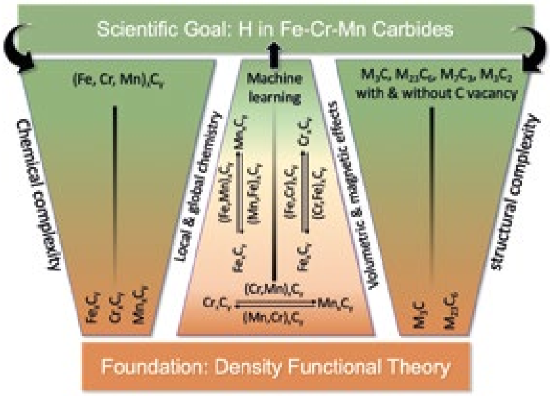

Fig. 4: *Research strategy for exploring chemical trends in the solubility of H in complex Fe-Cr-Mn carbides taking the chemical and structural complexity into account. The employed machine-learning approach is based on fundamental insights obtained from density functional theory.*

The computational design of functional materials was on the one hand devoted to hard-magnetic alloys. Using the example of $CeFe_{11}Ti$ revealed that the high-throughput optimization of certain key descriptors needs to be combined with an analysis of the phase stability of these alloys [45]. Based on these insights, we have analysed in an upcoming work, how this stability can be influenced by alloying elements (H. Sözen). On the other hand, a similar approach has been applied to Fe-based superconductors. Here, the delicate interplay of magnetic and van-der-Waals interactions have been used to describe the structural stability and the consequences for superconductivity in FeSe alloys [49]. This provided us the chance to systematically explore the impact of alloying elements such as S and Te on these properties (F. Lochner). In this way, we were able to demonstrate the relevance of our thermodynamic methods for novel materials and innovative applications.

Defect Chemistry and Spectroscopy (C. Freysoldt)

The group "Defect Chemistry and Spectroscopy" works on atomic-scale simulations of electrical charging phenomena by means of ab initio methods, and develops state-of-the-art methods and concepts for this purpose. Additionally, the group is growing towards machine-learning from the rich data sets produced by the key experiments at MPIE, notably electron microscopy and atom probe tomography.

The performance of functional energy materials - optoelectronics, solar cells, power electronics, batteries etc. - is intimately linked to where the electrical charges are stored at the atomic scale, and which processes are enabled or hindered at the relevant sites. Ab initio electronic structure theory is the decisive work horse for predictive simulations, but very challenging in practice due to the coupling of electronic effects at the local scale with macroscopic fields created far away, or artificially introduced by the modelling approach. While the group had long concentrated on the role of point defects in bulk materials, this has been gradually broadened to include surface- and interface effects, which are often of central importance in devices. This long-standing expertise is shared with other groups (Todorova, Wippermann) to support their innovations in electrochemical simulation techniques [15; see p. 69].

In the area of point defects, the group engages in various international collaborations. The recently developed charge correction scheme for surfaces and interfaces [50] has enabled investigations of defects in 2D materials in collaboration with R. Hennig (University of Florida), notably in MoS_2 [65]. Based on an initiative by Y. Kumagai (Tokio Institute of Technology), the charge correction scheme for bulk defects was further extended to vertical excitations in ionic materials [51]. The group's reputation is further underlined by the fact that several students from abroad recently secured funding for short- and long-term visits, which will take place once the pandemic situation permits.

The simulations of surfaces under extreme electric fields, such as encountered in atom probe tomography (APT) or field ion microscopy, have now been firmly established. M. Ashton, Alexander von Humboldt awardee, not only revealed a field-controlled two-stage evaporation path in field evaporation [28] (Fig. 5), but also addressed the puzzling behaviour of H in APT. The group maintains close bonds to the experimental APT group (B. Gault) and aims at deepening the understanding of field evaporation mechanisms from a DFT perspective (S. Katnagallu, since July 2021), as well as establishing an ab initio theory of field ion microscopy (S. Bhatt, PhD student since May 2021).

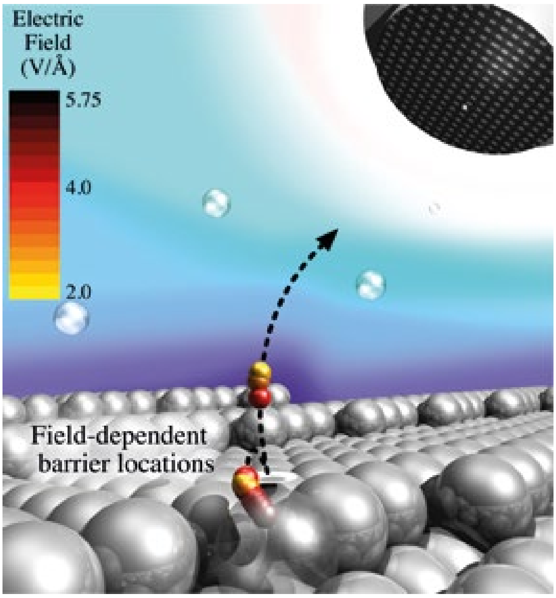

Fig. 5: *Visualization of field-controlled two-stage field evaporation paths of tungsten [28]. The red balls visualize the barrier position at different applied fields. A kink site atom may first roll up the step (lower set of barriers), and then evaporate from the step-position (upper part). At fields above 4 V/Å, the upper barrier vanishes and the kink atom undergoes direct evaporation from its original position.*

All these method developments would not be possible without the SPHInX package [84] as the code framework, which continues to be enhanced by the group. In addition to strengthening the integration with pyiron to exploit the latest features of the SPHInX DFT code, a complete rewriting of the algebraic core classes and partial reorganization of the physics code base has been undertaken in 2020 to overcome existing limitations, improve the maintainability, adopt a modern C++ standard, and simplify future extensions. This was released as SPHInX 3.0. In this context, significant efficiency gains for the k.p module could be realized (collaboration with O. Marquardt, Weierstraß-Institut Berlin).

As a new direction, triggered by the Max Planck network on Big-data driven material science (BiGmax, see p. 82), the group has started to explore machine-learning from the huge experimental data sets produced in electron microscopy and atom probe tomography. The unique opportunities coming from data-rich, yet noisy experimental signals that exhibit recurrent, but fuzzily defined patterns will require automated analysis tools that exploit material science domain knowledge. In electron microscopy (see p. 222), an efficient and robust segmentation of atomic-resolution images and videos, based on crystal symmetry descriptors was developed (N. Wang) [30]. Moreover, by adopting the idea of physics-informed neural networks it was demonstrated that a coarse-grained, very compact phase-field representation can be extracted from in-situ STEM time series (video sequences) of phase transformations and evolution (N. Wang, now L. Sreekala).

These activities have been further extended in the framework of the newly established Helmholtz Data Science School for Life, Earth, and Energy (HDS-LEE), which MPIE has joined as a partner institution. Embedded in the institute‘s strategy to push forward sustainable metallurgy, the first project aims at designing scrap-compatible Al alloys to increase the fraction of recycled Al, thus leveraging CO_2 emission from Al production from primary sources. The fate of unintentional alloying elements from unsorted scrap and the impact on mechanical performance is based on data mining from thermodynamic databases as well as from atom probe tomography. For the latter, a largely automatized workflow for detailed precipitate analysis was established (A. Saxena) – and immediately found applicable to entirely different material classes (Fig. 6).

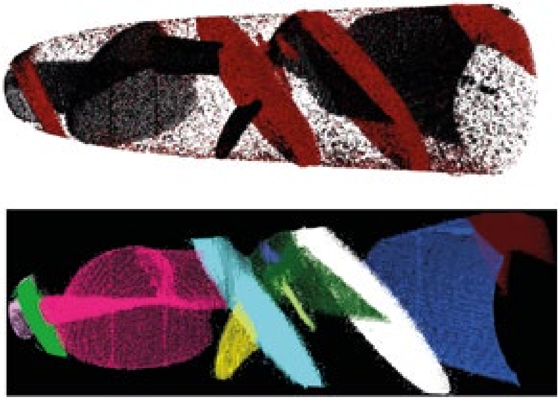

Fig. 6: *Automatic identification of precipitate phases via clustering in compositional space (upper image: black, red) in an APT data set from Fe-doped SmCo magnets, and subsequent segmentation into individual precipitates via DB-SCAN. APT data from N. Polin (MA).*

Thermodynamics and Kinetics of Defects (L. Huber)

The “Thermodynamics and Kinetics of Defects (TKD)” group headed by L. Huber since 2019 focuses on the behaviour of defects -- grain and phase boundaries, dislocations, and point-defects at the atomic scale using classical, quantum, and machine learned representations. An example of this work, a defect phase diagram, is shown in Fig. 7. Since its inception, the group has had two MSc students graduate: R. Dsouza studying the impacts of anharmonicity on diffusion (“Fully anharmonic self-diffusion coefficients using the Finite Temperature String method”, RWTH Aachen, Dec. 2020), and V. Bhuva studying atomic clustering behaviour ("Algorithms for optimal chemical ordering ", Universität Passau, Sept. 2021). R. Dsouza has continued in the group pursuing his doctorate with a focus on anharmonic contributions to free energies. In Feb. 2020 M. Poul also joined the group as a PhD student. His work lies within our collaborative research centre CRC1394 with RWTH Aachen (see p. 76) and is on developing machine learned potentials suitable for complex phases and solute-defect interactions. The scope of research activities within the TKD group is given more completely on page (see p. 41).

Members of the TKD group are also heavily involved in the development of the CM department's open-source pyiron IDE (see p. 72). In September 2020 the group hired N. Siemer as a full time software developer. As part of his work to bring pyiron beyond atomistic simulation and make it relevant for our experimentalists, he has worked on a graphical user interface (GUI) for pyiron and collaborated with our experimentalist colleagues to develop comprehensive metadata schemes for digitalizing over 22 different experimental activities, e.g. TEM measurements, tensile tests, and various sample preparation routines. L. Huber and N. Siemer, together with M. Poul, were also heavily involved in the evolution of pyiron from a single monolithic package to a collection of modules, each with its own specialization and all with state-of-the-art continuous integration/continuous development (CI/CD) workflows. Since this split the TKD group has contributed to various specialized modules, e.g. incorporating finite element codes to bring pyiron into the regime of continuum mechanics (L. Huber), and powerful streamlining for the architecture pyiron uses for data storage and retrieval (M. Poul). R. Dsouza and V. Bhuva's scientific work is also fully integrated with the pyiron environment. In particular, R. Dsouza along with L. Huber and a collaborator at Montan Universität Leoben developed a generic scheme for workflows on directed cyclic graphs.

In addition to digital infrastructure, TKD members are involved in a variety of organizational and social infrastructure activities. R. Dsouza and M. Poul represent the CM department to MPIE's PhD student board, and L. Huber serves on the research data management team for CRC1394 and MPIE TeamGreen (see p. 103). M. Poul has also been particularly active in workshop organization, helping to coordinate and execute both the RUB ICAMS POTENTIAL workshop [85] and a pyiron workshop on calculating phase diagrams [86]. M. Poul and R. Dsouza also acted as teaching assistants in J. Neugebauer's recurring electronic structure course and L. Huber facilitated a series of hands-on simulation sessions for a graduate level course at RWTH Aachen in the winter semester of 2020/21.

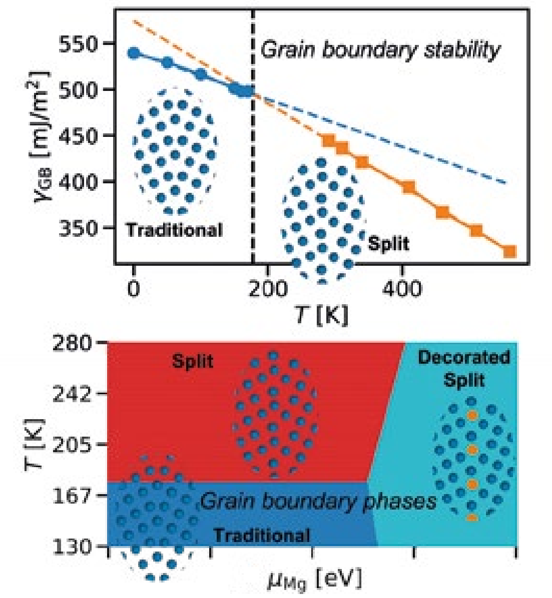

Fig. 7: *Top: A grain boundary phase transition in Al by classical molecular statics. Bottom: An simplified defect phase diagram for the same boundary in an Al-Mg alloy as function of temperature and Mg chemical potential.*

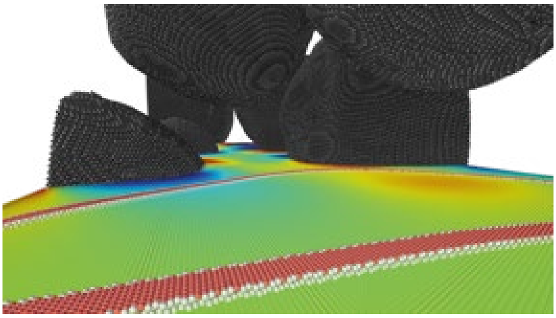

Fig. 8: *Superdislocation interacting with precipitates obtained by APT of a superalloy. The colour represents the resolved shear stress acting on the superdislocation caused by the lattice misfit of the precipitates, which can affect the strengthening mechanism by decoupling the superpartial dislocations [87].*

Microstructure and Mechanics (E. Bitzek)

The new group of E. Bitzek (see p. 37) on "Microstructure and mechanics" ideally complements the expertise already present at CM and strengthens the departments activities regarding mechanical properties and failure of materials. With its background and its methods, the new group can directly contribute to research throughout the departments, as has been already demonstrated by several collaborations. Collaborations with groups working on high entropy alloys and concentrated solid solutions, high temperature materials, hydrogen in materials, nanomechanics and nanostructured materials are expected to lead to synergies with the microstructure and mechanics group. Additionally, the new group broadens the scope of MPIE by including new classes of materials like bulk oxide, polymer or metallic glasses, and strengthens its activities in research data management and the NFDI initiative.

Electrochemistry and Corrosion (M. Todorova)

The “Electrochemistry and Corrosion” group aims at understanding how interactions with the environment affect and modify materials and their properties. Hereby, the strong focus on corrosion and electrochemical processes, where solid/liquid interfaces play a central role, continuously challenges the use of conventional techniques and drives us to extend existing methods and/or develop new approaches, enabling us to gain an in-depth atomistic understanding of the physical and chemical processes taking place at surfaces and interfaces (Fig.9). Particularly fruitful in this context was the close collaboration with the groups of S. Wippermann (GO) and C. Freysoldt (CM) leading to the development of a novel thermopotentiostat scheme (see p. 69), which allows for a realistic description of electrified solid/liquid interfaces [15].

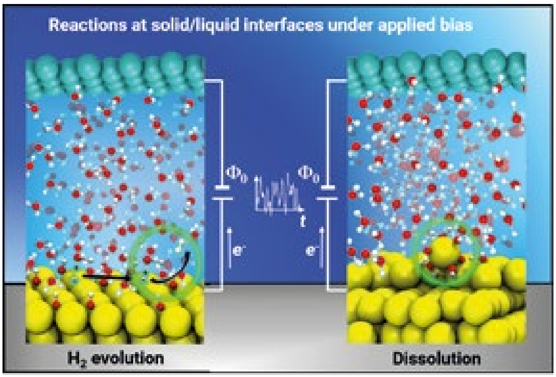

Fig. 9: *Reactions at solid/liquid interfaces under realistic conditions of applied bias. The voltage is applied between two electrodes (yellow and cyan balls), which sandwich the water electrolyte (oxygen: red, hydrogen: white). Calculated reactions and reaction paths occurring at the interface are highlighted. Left: H_2 evolution on Pt(111). Right: Mg dissolution under anodic conditions.*

Exploring the connection between applied potential and water interactions with solid surfaces the work of S. Surendralal [16] provided fascinating insights into the H/H_2O/Pt(111) system und led us to re-evaluate existing models of water-metal bonding, Fig. 10 (see p. 224). We continue with this work by evaluating the energetics of the system, investigating both free energy profiles gained from the performed ab initio molecular dynamics calculations, as well as the impact a change in the hydrogen chemical potential has on the H coverage at the surface. Recently, the work was extended to the consideration of a more open surface orientation Pt(100) by Z. Wang. In both cases, we closely collaborate with the group of Prof. R. K. Campen (University Duisburg-Essen) exploring spectroscopic signatures to gain a better understanding of mobility and reactivity at and of the surface.

The award of a FP-RESOMUS fellowship (see p. 86) to Z. Wang enables him to address very fundamental questions related to charge transfer reactions, which are at the heart of any electrochemical process, at solid/liquid interfaces. Using model systems, he explores, for example, the interaction between solvation shells or the change in water screening properties during a dissolution process.

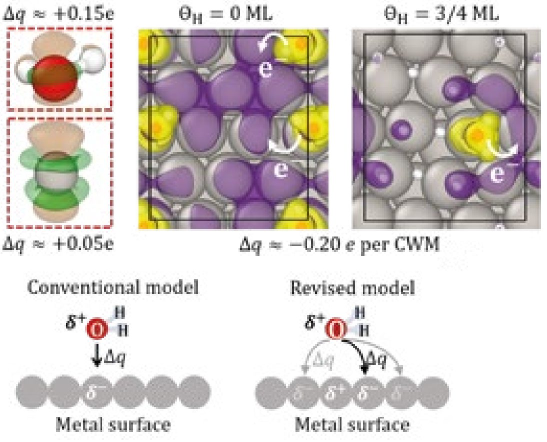

Fig. 10: *Top: Charge density difference showing the charge redistribution in the H/Pt(111)/H_2O system. Side view of the charge distribution of a water molecule chemisorbed on top of a Pt atom with respective charges Dq obtained by integration over the volumes indicated by the red dashed lines (left). Top view of the charge redistribution for different coverages of co-adsorbed H (middle, right). Bottom: Conventional model for chemisorbed water-metal bonding (left) and revised model, based on our findings (right). Adapted from [16].*

Further work concerned with the transfer of electrons in the context of electrochemical reactions was carried out in collaboration with the group of D. Marx (Ruhr-Universität Bochum) within the Cluster of Excellence RESOLV. S.Yoo utilised alignment techniques commonly used in semiconductor physics, to identify the mutual position of relevant energy levels with respect to each other. This allowed elucidating the role of electron transfer between a surface oxygen vacancy on TiO_2 and an adsorbed Au nano-cluster, highlighting the importance of considering the dynamic nature of the cluster and providing fascinating insights into the catalytic activity and reactivity of this system [17].

During our investigations related to the role of point defects in the growth of oxide barrier layers during corrosion, we realised that materials like ZnO, important in the context of the corrosion protection of steels, suffer some limitations in the description of their electronic structure. S. Yoo tracked this back to the presence of spontaneous polarizations, which is not accounted for by conventional passivation schemes used when modelling semiconductor surfaces. This led us to develop in collaboration with Prof. C. Van de Walle (UCSB, USA) a robust generalized passivation method that accounts for the effect of spontaneous polarization for pyroelectric materials and correctly describes the asymptotic bulk limit [46].

While ab initio techniques are a prerequisite to obtain an accurate description of the electronic structure during processes triggered by interactions with the environment, they impose restrictions like, e.g., system size or achievable time scales. Aiming to overcome these and other limitations, and further improve the realism of our models in describing processes at electrified solid/liquid interfaces, we closely collaborate with the group of S. Wippermann (GO). During weekly hackathon sessions we explore various ideas and directions aiming to overcome such limitations, and/or explore new directions.

The use of thermodynamic concepts and various flavours of defect phase diagrams have always been an integral part of the activities in the "Electrochemistry and Corrosion"group. The establishment of the CRC1394 (see p. 76) has recently intensified these activities, which are carried out in close collaboration with the groups of C. Scheu (SN), T. Hickel (CM) and L. Huber (CM). S.K. Kanachari Bavajigari focuses on the solid solution of Ca and Al in a Mg matrix. Constructing surface and interface Pourbaix diagrams, i.e. thermodynamic diagrams providing information about the stability of phases depending on the electrode potential U and the pH of the solution, he explores the connection to the chemical potential variations of the involved species. His recently obtained Alexander von Humboldt postdoctoral fellowship will allow him to explore the atomistic mechanism of the role of Fe in Mg corrosion. E.I. Saad, who looks into routes for efficient free energy calculations, supports these activities.

Thermodynamic modelling and surface phase diagrams have also proven extremely valuable during the course of recent collaborations with the group of B. Gault (MA). They enabled us to explain the role of H as a contaminant in Atom probe experiments [59] (see p. 198) or account for different conditions employed to synthesise Pd nanoparticles. In close collaboration with the groups of B. Gault (MA), T. Hickel (CM), C. Scheu (SN) and O. Kasian (GO) we were able to gain amazing insights into the contamination of nanoparticles during synthesis [18] and its impact on the catalytic activity of such particles (see p. 198 & 184).

Growth Modelling project group (L. Lymperakis)

The growth modelling project group focuses on the epitaxial growth and the properties of semiconductors, nanostructures, and 2D heterostructures by means of ab-initio based simulations. Within the reporting period L. Lymperakis is Co-PI of an EU and BMBF co-funded project (UltimateGaN: Research for GaN technologies, devices and applications to address the challenges of the future GaN roadmap) and a DAAD (Deutscher Akademischer Austauschdienst) funded project (Strain Tuning of III-V Semiconductor Nanowires, TUNE). Topics addressed by the growth modelling group include, but are not limited to surfaces and extended defects in materials for power- and opto- electronic applications, synthesis and properties of novel III-Nitride ternary alloys, and interfaces in Silicon.

Group III-Nitrides constitute one of the most important families of semiconductor materials with technological applications in the fields of energy efficient optoelectronic and power electronic applications. Crystal defects, in particular dislocations constitute a long-standing controversial topic in these materials. Although highly efficient optoelectronic devices operate at dislocation densities as high as 10^8 to 10^{10} cm^{-2}, GaN-based power electronics, a key component in future smart grid power applications, is severely affected by them. Dislocation and associated V-pits, i.e., structural defects which are formed when dislocations pin a surface or an interface, have been proposed to be the root cause of device leakage and breakdown and are considered to be one of the major limiting factors in achieving the full potential of nitride-based power electronic devices. An intriguing feature of these defects is that experimental evidences suggest that they are associated with open core structures, thus forming nanopipes with diameters of a few nms.

The formation of both nanopipes and V-pits is the result of a complex interplay between strain energy and surface energies. A necessary prerequisite to control the formation and the properties of these defects is to derive the surface energies of low index semipolar and nonpolar planes with respect to the energy of the (0001) GaN surfaces. In a first step, S. Yoo developed a novel reconstruction inspired passivation scheme that allows to unambiguously investigate the energetics and electronic properties of semipolar surfaces of semiconductors with a singular polar axis [52]. This scheme is generic and robust and can be straightforwardly employed in first principles investigations of low-symmetry surfaces as well as in high-throughput and/or machine learning studies. Based on this scheme S. Yoo calculated the surface energies of low index GaN planes and constructed a Wulff diagram as well as a 'V-pit' phase diagram, which describes the equilibrium size and shape of V-pits as a function of the ambient growth conditions. This diagram reveals that under typical growth conditions, the GaN polar planes are intrinsically unstable against the formation of V-pits with equilibrium lateral sizes up to a few tenths of a nm at sites where screw threading dislocations reach the surface. The defect formation is driven by the preferential decoration of the surfaces by hydrogen, which reduces the surface energies.

The formation and the properties of dislocation induced nanopipes in III-nitrides was investigated by combining first principles with large scale empirical potential calculations (see p. 190). Based on these calculations we constructed a dislocations' phase diagram which describes the energetically most favorable core structures as function of the growth conditions. A general trend that emerges from the aforementioned phase diagram is that nanopipes are energetically favorable under technologically relevant growth conditions. Although nanopipes exhibit large free surface areas which increase the energy, they can be energetically favorable if the surface energy is compensated by the core and strain energy of the material removed to create the open core dislocation. Under the aforementioned conditions the cation and anion dangling bonds at the open core inner surfaces are passivated by NH_x molecules and H atoms, respectively. This reduces considerably the surface energy and the formation of nanopipes with hexagonal cross section and equilibrium diameters ranging for ≈1 to ≈2 nm is energetically favorable. The formation of nanopipes has important implications for the efficiency and reliability of power electronic devices: Nanopipes and the associated inner surfaces constitute efficient paths for impurities diffusion and offer high density of active sites for incorporation.

Using its strong expertise in ab initio techniques and compound semiconductors, the group was instrumental in performing a wide range of joint computational and experimental studies. Examples are studying the optoelectronic properties and carrier confinement in InGaN quantum structures [66], strain engineering to achieve high In content InGaN Quantum Wells [67] (in collaboration with the Leibniz Institute for Crystal Growth, Berlin), the substitutional synthesis of sub-nanometer InGaN/GaN quantum wells [68] (in collaboration with the Aristotle University of Thessaloniki, Greece and the Helmholtz-Zentrum Dresden-Rossendorf, Dresden), the investigation and design of novel $Al_{5+\alpha}Si_{5+\delta}N_{12}$ compounds [69] (in collaboration with the Université Côte d'Azur, CRHEA-CNRS, France), or unravelling the mechanism leading to semi-insulating Carbon doped GaN [70] (in collaboration with Infineon Technologies Austria).

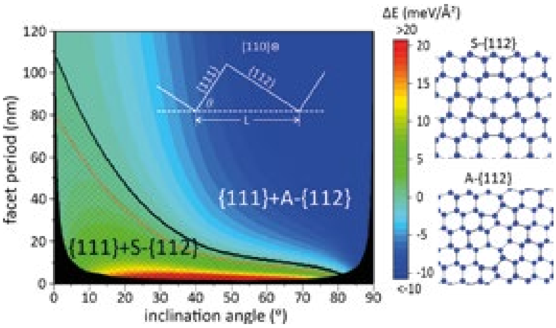

Fig 11: *Interface phase diagram showing the most stable interface reconstructions and orientation (faceting) as function of the GB inclination angle in silicon. The solid black curve indicates the boundary between GBs having an asymmetric (A) - or symmetric (S)-{112} facet. The cross-hatched area denotes the stability region of the higher energy S-{112} facets. The black shaded area marks the region of geometrically inaccessible facet periods and inclination angles. Inset: Schematic representation of a Σ3 tilt GB (dashed line) inclined with respect to {111} faceted toward {111} and {112} facets. Right panel: Relaxed atomic geometry of the S-{112} and the A-{112} GB viewed along ⟨110⟩. Adapted from [21]*

Applying its deep expertise on surfaces the group extended its activities in the investigations of interfaces in polycrystalline materials. In multi- and polycrystalline Si, the base material in today's photovoltaic industry, grain boundaries (GB) severely impact device performance. GB faceting severely impacts their behaviour. The mechanisms underlying faceting of GBs in Si were investigated by employing DFT and MEAM (Modified Embedded Atom Method) potential calculations [21]. The picture deduced by our calculations contradicts the common perception that the properties of faceting are merely driven by the anisotropic GB energies. Although anisotropic GB energies are a prerequisite for GB faceting, higher energy metastable GB phases are stabilized by thermodynamics and not kinetics when constituting the facets at line junctions (Fig 11). This insight has implications for the design of electronic and structural materials: Microstructures that allow for large facet periods stabilize line junctions accommodated by dislocations. In contrast, fine granular structures that limit facet periods promote line junctions without extended line defects and long-range strain fields.

Complex Concentrated Alloys (CCA) project group (F. Körmann)

The "Complex Concentrated Alloys (CCA)" research group of Fritz Körmann, established back in 2018 (see p. 55), aims in particular at the method development and application to complex concentrated alloys [8] including the prominent class of high entropy alloys (see p. 210, p. 194, p. 208).

Machine learning potentials – method development

A key achievement within the last years has been the combination of modern machine learning interatomic potentials (MLIPS) with ab initio calculations [1-4]. Two kinds of MLIPS have been applied to the challenging class of multicomponent alloys. One is the low rank potentials [1, 3, 4], a highly efficient on-lattice potential, suitable to explore, in combination with Monte Carlo simulations chemical orderdisorder transitions in multi-component alloys [1, 3, 4]. It can account for local lattice relaxations, which can qualitatively alter the computationally predicted ordered phases [1]. Short-range order, which impact on materials properties is currently attracting significant attention, can be readily computed. Applications include prototypical bcc NbMoTaW and AlNbTiV alloys [1,4] as well as recently discovered [73] ultra-strong fcc VCoNi alloys [3].

The second type of MLIPS are the moment tensor potentials (MTPs), fitting accurately energies, forces and stresses. Based on thermodynamic integration, highly accurate free energies, including anharmonic contributions can now be computed for multicomponent alloys, outperforming previous empirical potentials [2]. In combination with active learning [41] and novel structural descriptors [74], these potentials can be used to explore wide composition and temperature ranges to predict temperature-compositional phase diagrams such as the omega-bcc-liquid stability in bcc TiZrHfTa alloys [41].

Selected applications

The vast compositional phase space and chemical complexity of multicomponent alloys offers possibilities to

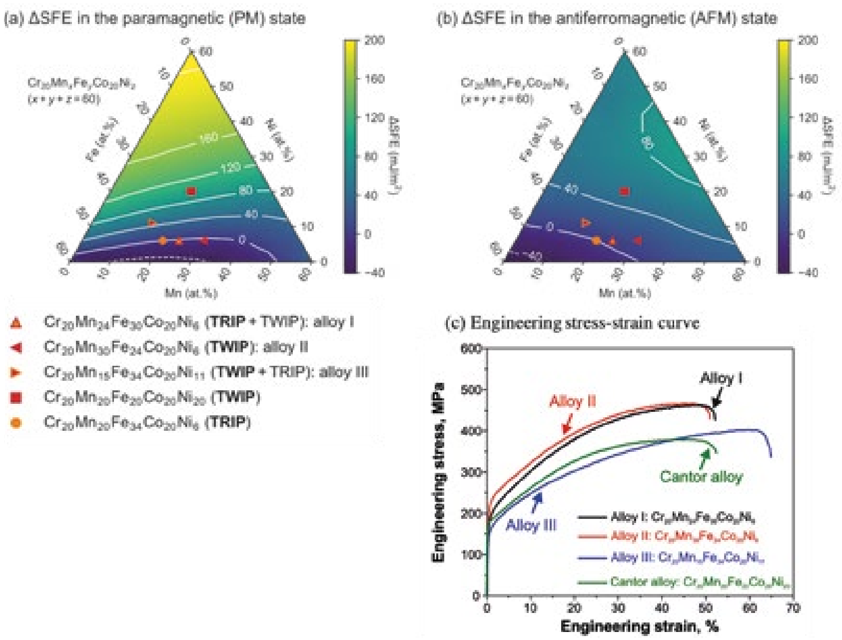

Fig. 12: *(a), (b) Difference in the Stacking Fault Energies (SFEs) of quinary* $Cr_{20}Mn_xFe_yCo_{20}Ni_z$ *CCAs compared to a reference alloy* $Cr_{20}Mn_{20}Fe_{34}Co_{20}Ni_6$ *in the (a) paramagnetic and in the (b) antiferromagnetic states obtained from ab initio calculations [75]. Triangle symbols show the experimentally investigated compositions. Two reference alloys, namely, the Cantor alloy (square) and* $Cr_{20}Mn_{20}Fe_{34}Co_{20}Ni_6$ *(circle), which is known to show transformation induced plasticity (TRIP), are also shown. The colors of the symbols show the dominant deformation behavior of the corresponding compositions; orange and red are for TRIP and twinning induced plasticity (TWIP), respectively. (c) Comparison of mechanical responses of three investigated alloys with Cantor alloy.*

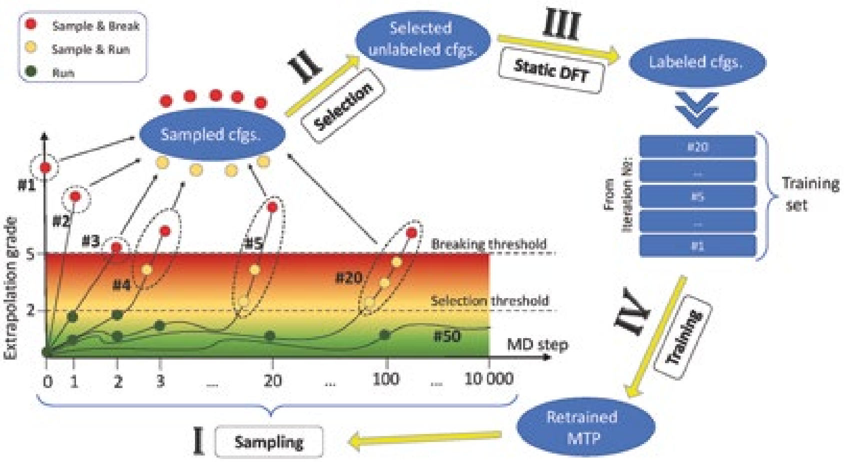

Fig. 13: *Graphical representation of the active learning schema used to design Machine Learning Moment Tensor Potentials for bcc multicomponent alloys [41]. (I) Configurations (cfgs.) are sampled from MD trajectories (II) Sampled configurations are shortlisted based on the MaxVol criterion [41]. (III) After assigning DFT energies/forces/ stresses they are appended to the training set. (IV) Training of a new MTP able to "work" in larger configurational space. This procedure is repeated until no new configurations are sampled from the MD trajectory.*

tune alloy composition for improving intrinsic materials properties. Key ingredients in this respect are, e.g., local lattice distortions and atomic level pressures [7, 10 ,12] as well as stacking fault energies [75, 79, 80] (see p. 194 & p. 208). Based on high-throughput calculations and careful consideration of magnetic effects, the intrinsic interplay of magnetic entropy and chemical composition has been revealed for Cantor based fcc alloys, allowing to develop in cooperation with our experimental colleagues a number of new transformation and twinning induced plasticity high entropy alloys [75]. Interstitial alloying introduces a further challenge due to the immense phase space of local chemical environments. New thermodynamic statistical methods were developed to account for this allowing to predict the impact of interstitial elements for fine-tuning the alloys stacking fault energies [5, 13, 81]. Further opportunities are in the field of precipitation-strengthened CCAs [14, 82]. To improve the magnetic properties tuning with non-magnetic elements [82] as well as spinodal composition has been introduced as design concepts for CCAs [76] showing that in combination with ab initio predicted energetics, the magnetic properties such as Curie temperature and saturation magnetization can be readily improved (see p. 210). Also surface stability of CCAs has attracted enormous attention recently as potential game changers in heterogeneous catalysis, showing much higher catalytic activities as simple rule of mixture would suggest. Recent research efforts are therefore also devoted to study surface segregation and stability of various CCAs [22, 9].

References

1. Kostiuchenko, T.; Körmann, F.; Neugebauer, J.; Shapeev, A.: npj Comput. Mater. 5 (2019) 55.
2. Grabowski, B.; Ikeda, Y.; Srinivasan, P.; Körmann, F.; Freysoldt, C.; Duff, A.; Shapeev, A.; Neugebauer, J.: npj Comput. Mater 5 (2019) 80.
3. Kostiuchenko, T.; Ruban, A.V.; Neugebauer, J.; Shapeev, A.; Körmann, F.: Phys. Rev. Mater. 4 (2020) 113802.
4. Körmann, F.; Kostiuchenko, T.; Shapeev, A.; Neugebauer, J.; Phys. Rev. Mater. 5 (2021) 053803.
5. Ikeda, Y.; Tanaka, I.; Neugebauer, J.; Körmann, F.: Phys. Rev. Mater. 3 (2019) 113603.
6. Ikeda, Y., Grabowski, B., Körmann, F., Mater. Charact. 147 (2019) 49.
7. Oh, H.S., Kim, S.J., Odbadrakh, K., Ryu, W.H., Yoon, K.N., Mu, S., Körmann, F., Ikeda, Y., Tasan, C.C., Raabe, D., Nat. Comm. 10 (2019) 8.
8. Ferrari, A., Dutta, B., Gubaev, K., Ikeda, Y., Srinivasan, P., Grabowski, B., Körmann, F., J. Appl. Phys. 128 (2020) 150901.
9. Ferrari, A., Körmann, F., Appl. Surf. Sci. 533 (2020) 147471.
10. Ishibashi, S., Ikeda, Y., Körmann, F., Grabowski, B., Neugebauer, J., Phys. Rev. Mater. 4 (2020) 023608.
11. Körmann, F., Li, Z., Raabe, D., Sluiter, M.H.F., Iron-rich High Entropy Alloys, in: R. Rana (Ed.) High-Performance Ferrous Alloys, Springer, Cham2021, pp. 32.
12. Oh, H.S.; Odbadrakh, K.; Ikeda, Y.; Mu, S.; Körmann, F.; Sun, C.; Ahn, H.S.; Yoon, K.N.; Ma, D.; Tasan, C.C.; Egami, T.; Park, E.S.: Acta Mater. 216, (2021) 117135.
13. Ikeda, Y.; Körmann, F.: J. Phase Equilib. Diffus. 42, (2021) 551–560.
14. Jang, T.K.; Choi, W.S.; Kim, D.W.; Choi, G.; Jun, H.; Ferrari, A.; Körmann, F.; Choi, P.; Sohn, S.S.: Nat. Commun. 12, (2021) 4703.
15. Deißenbeck, F.; Freysoldt, C.; Todorova, M.; Neugebauer, J.; Wippermann, S.: Phys. Rev. Lett. 126 (2021) 136803.
16. Surendralal, S.; Todorova, M.; Neugebauer, J.: Phys. Rev. Lett. 126 (2021) 166802.
17. Yoo, S.-H.; Siemer, N.; Todorova, M.; Marx, D.; Neugebauer, J.: J. Phys. Chem. C 123 (2019) 5495.
18. Kim, S.-H.; Yoo, S.-H.; Chakraborty, P.; Jeong, J.; Lim, J.; El-Zoka, A.; Zhou, X.; Stephenson, L. ; Hickel, T.; Neugebauer, J.; Scheu, C.; Todorova, M.; Gault, B.: (submitted), https://arxiv.org/abs/2106.06342
19. Korte-Kerzel, S.; Hickel, T.; Huber, L.; Raabe, D.; Sandlöbes-Haut, S.; Todorova, M.; Neugebauer, J.: Int. Mater. Rev., https://www.tandfonline.com/doi/full/10.1080/09506608.2021.1930734
20. Šlapáková, M.; Zendegani, A.; Liebscher, C.H.; Hickel, T.; Neugebauer, J.: Hammerschmidt, T.; Ormeci, A.; Grin, J.; Dehm, G.; Kumar, K.S.; Stein, F.: Acta Mater. 183 (2020) 362.
21. M. Alam, M.; Lymperakis, L.; Neugebauer, J.: Phys. Rev. Mater. 4 (2020) 083604.
22. J. Guénolé, F.-Z. Mouhib, L. Huber, B. Grabowski, S. Korte-Kerzel, Scripta Mater., 166 (2019) 5
23. Darvishi Kamachali, R., Kwiatkowski da Silva, A., McEniry, E., Ponge, D., Gault, B., Neugebauer, J., Raabe, D., npj Comput. Mater. 6 (2020) 191.
24. Zhao, H., Huber, L., Lu, W.J., Peter, N.J., An, D.Y., De Geuser, F., Dehm, G., Ponge, D., Neugebauer, J., Gault, B., Raabe, D., Phys. Rev. Lett. 124 (2020) 106102.
25. Ko, W.-S., Stukowski, A., Hadian, R., Nematollahi, A., Jeon, J.B., Choi, W.S., Dehm, G., Neugebauer, J., Kirchlechner, C., Grabowski, B., Acta Mater. 197 (2020) 54.
26. Zhang, X.; Wang, H.; Hickel, T.; Rogal, J.; Li, Y.; Neugebauer, J.: Nature Mater. 19 (2020) 849.
27. Alam, M., Lymperakis, L., Groh, S., Neugebauer, J., Model. Sim. Mater. Sci. Eng.; https://doi.org/10.1088/1361-651X/ac3a15
28. Ashton, M.; Mishra, A.; Neugebauer, J.; Freysoldt, C.: Phys. Rev. Lett. 124 (2020) 176801.
29. Ferraz Morgado, F.; Katnagallu, S.; Freysoldt, C.; Klaes, B.; Vurpillot, F.; Neugebauer, J.; Raabe, D.; Neumeier, S.; Gault, B.; Stephenson, L.T.: Scripta Mater. 203 (2021) 114036.
30. Wang, N.; Freysoldt, C.; Zhang, S.; Liebscher, C.H.; Neugebauer, J.: Microsc. Microanal. (submitted); https://doi.org/10.1017/S1431927621012770
31. Katnagallu, S., Stephenson, L.T., Mouton, I., Freysoldt, C., Subramanyam, A.P.A., Jenke, J., Ladines, A.N., Neumeier, S., Hammerschmidt, T., Drautz, R., Neugebauer, J., Vurpillot, F., Raabe, D., Gault, B., New J. Phys. 21 (2019) 123020.
32. Janssen, J., Surendralal, S., Lysogorskiy, Y., Todorova, M., Hickel, T., Drautz, R., Neugebauer, J., Comp. Mater. Sci. 163 (2019) 24.
33. Lysogorskiy, Y., Hammerschmidt, T., Janssen, J., Neugebauer, J., Drautz, R., Model. Sim. Mater. Sci. Eng. 27 (2019) 025007.
34. Janssen, J., Makarov, E., Hickel, T., Shapeev, A.V., Neugebauer, J., npj Comput. Mater. (submitted); http://arxiv.org/abs/2112.04081
35. Schaarschmid, J., Yuan, J., Strunk, T., Kondov, I., Huber, S.P., Pizzi, G., Kahle, L., Bölle, F.T., Castelli, I.E., Vegge, T., Hanke, F., Hickel, T., Neugebauer, J., Caldeira Rego, C.R., Wenzel, W., Adv. Energy Mater. (2021), in print.
36. Zhu, L.-F., Janssen, J., Ishibashi, S., Körmann, F., Grabowski, B., Neugebauer, J., Comp. Mater. Sci. 187 (2021) 110065.
37. Dutta, B.; Körmann, F.; Ghosh, S.; Sanyal, B.; Neugebauer, J.; Hickel, T.: Phys. Rev. B 101 (2020) 094201.
38. Bigdeli, S.; Zhu, L.-F.; Glensk, A.; Grabowski, B.; Lindahl, B.; Hickel, T.; Selleby, M.: Calphad 65 (2019) 79.
39. Gupta, A.; Tas, B.; Korbmacher, D.; Dutta, B.; Neitzel, Y.; Grabowski, B.; Hickel, T.; Esin, V.; Divnninski, S.V.; Wilde, G.; Neugebauer, J.: Materials 14 (2021) 1837.
40. Hegde, O.; Grabowski, M.; Zhang, X.; Waseda, O.; Hickel, T.; Freysoldt, C.; Neugebauer, J.: Phys. Rev. B 102 (2020) 144101.
41. Gubaev, K.; Ikeda, Y.; Tasnádi, F.; Neugebauer, J.; Shapeev, A.V.; Grabowski, B.; Körmann, F.: Phys. Rev. Mater. 5 (2021) 073801.
42. Swinburne, T.D., Janssen, J., Todorova, M., Simpson, G., Plechac, P., Luskin, M., Neugebauer, J., Phys. Rev. B 102 (2020) 100101(R).
43. Zhu, L.-F., Körmann, F., Ruban, A.V., Neugebauer, J., Grabowski, B., Phys. Rev. B 101 (2020) 144108.

44. Hegde, O., Kulitckii, V., Schneider, A., Soisson, F., Hickel, T., Neugebauer, J., Wilde, G., Divinski, S., Fu, C.-C., Phys. Rev. B (2021), Phys. Rev. B 104 (2021) 184107.

45. Sözen, H.I.; Ener, S.; Maccari, F.; Skokov, K.P.; Gutfleisch, O.; Körmann, F.; Neugebauer, J.; Hickel, T.: Phys. Rev. Mater. 3 (2019) 084407.

46. Yoo, S.-H.; Todorova, M.; Wickramaratne, D.; Weston, L.; Van de Walle, C.G.; Neugebauer, J.: npj Comput. Mater. 7 (2021) 58.

47. Freysoldt, C.; Mishra, A.; Ashton, M.; Neugebauer, J.: Phys. Rev. B, 102 (2020) 045403.

48. Glensk, A.; Grabowski, B.; Hickel, T.; Neugebauer, J.; Neuhaus, J.; Hradil, K.; Petry, W.; Leitner, M.: Phys. Rev. Lett. 123 (2019) 235501.

49. Lochner, F.; Eremin, I.M.; Hickel, T.; Neugebauer, J.: Phys. Rev. B 103 (2021) 054506.

50. Freysoldt, C.; Neugebauer, J: Phys. Rev. B 97 (2018) 205425.

51. Gake, T.; Kumagai, Y.: Freysoldt, C.; Oba, F.: Phys. Rev. B 101 (2020) 020102(R).

52. Yoo, S.-H.; Lymperakis, L.; Neugebauer, J.: Phys. Rev. Mater. 5 (2021) 044605.

53. Freysoldt, C., Merz, P., Schmidt, M., Mohitkar, S., Felser, C., Neugebauer, J., Jansen, M., Angew. Chem. Int. Edit. 58 (2019) 149.

54. Glensk, A., Grabowksi, B., Hickel, T., Neugebauer, J., Neuhaus, J., Hradil, K., Petry, W., Leitner, M., Phys. Rev. Lett. 123 (2019) 6.

55. Korbmacher, D., Glensk, A., Duff, A.I., Finnis, M.W., Grabowski, B., Neugebauer, J., Phys. Rev. B 100 (2019) 104110.

56. Kwiadkowksi da Silva, A., Kamachali, R.D., Ponge, D., Gault, B., Neugebauer, J., Raabe, D., Acta Mater. 168 (2019) 109.

57. Kratzer, P., Neugebauer, J., Front. Chem. 7 (2019) 106.

58. Maurer, R.J., Freysoldt, C., Reilly, A.M., Brandenburg, J.G., Hofmann, O.T., Björkman, T., Lebègue, S., Tkatchenko, A., Ann. Rev. Mater. Res. 49 (2019) 30.

59. Yoo, S.-H.: Kim, S.-H.; Woods, E.; Gault, B.; Todorova, M.; Neugebauer, J.; Neu J. Phys. (in press), https://doi.org/10.1088/1367-2630/ac40cd

60. Esakkiraja, N.; Gupta, A.; Jayaram, V.; Hickel, T.; Divinski, S.V.; Paula, A.: Acta Mater. 195 (2020) 35.

61. Mu, S.; Olsen, R.; Dutta, B.; Lindsay, L.; Samolyuk, G.D.; Berlijn, T.; Specht, E.D.; Jin, K.; Bei, H.; Hickel, T.; Larson, B.C.; Stocks, G.M.: npj Comput. Mater. 6 (2020) 4.

62. Schneider, A.: Fu, C.-C.; Waseda, O.; Barreteau, C.; Hickel, T.: Phys. Rev. B 103 (2021) 024421.

63. Palanisamy, D.; Kovács, A.; Hegde, O.; Dunin-Borkowski, R.E.; Raabe, D.; Hickel, T.; Gault, B.: Phys. Rev. Mater. 5 (2021) 064403.

64. Ashton, M., Trometer, N., Mathew, K., Suntivich, J., Freysoldt, C., Sinnott, S.B., Hennig, R.G., J. Phys. Chem. C 123 (2019) 8.

65. Tan, A.M.Z.; Freysoldt, C.; Hennig, R.G.: Phys. Rev. Mater. 4 (2020) 114002. – 2D

66. Anikeeva, M.; Albrech, M.; Mahler, F.; Tomm, J.W.; Lymperakis, L.; Cheze, C.; Calarco, R.; Neugebauer, J.; Schulz, T.: Sci. Rep. 9 (2019) 9047.

67. Schulz, T.; Lymperakis, L.; Anikeeva, M.; Siekacz, M.; Wolny, P.; Markurt, T.; Albrecht, M.: Phys. Rev. Mater., 4 (2020) 073404.

68. Vasileiadis, I. G.; Lymperakis, L.; Adikimenakis, A.; Gkotinakos, A.; Devulapalli, V.; Liebscher, C. H.; Androulidaki, M.; Hübner, R.; Karakostas, Th.; Georgakilas, A.; Komninou, Ph.; Dimakis, E.; Dimitrakopulos, G. P.: Sci. Rep. 11 (2021) 20606.

69. Dagher, R.; Lymperakis, L.; Delaye, V.; Largeau, L.; Michon, A.; Brault, J.; Vénnègues, P.: Sci. Rep. 9 (2019) 15907.

70. Koller, C.; Lymperakis, L.; Pogany, D.; Pobegen, G.; Ostermaier, C.: J. Appl. Phys. 130 (2021) 185702.

71. Kiranbabu, S.; Tung, P.-Y.; Sreekala, L.; Prithiv, T.S.; Hickel, T.; Pippan, R.; Morsdorf, L.; Herbig, M.: Mater. SCie. Eng. A (2021) 142372

72. Sözen, H.I.; Hickel, T.; Neugebauer, J.: Calphad 68 (2020) 101731.

73. Sohn, S.S.; Kwiatkowski da Silva, A.; Ikeda, Y.; Körmann, F.; Lu, W.; Choi, W.S.; Gault, B.; Ponge, D.; Neugebauer, J.; Raabe, D.: Adv. Mater. 31 (2019) 1807142.

74. Ikeda, Y.; Gubaev, K.; Neugebauer, J.; Grabowski, B.; Körmann, F.: npj Comput. Mater. 7 (2021) 34.

75. Wu, X.; Li, Z.; Rao, Z.; Ikeda, Y.; Dutta, B.; Körmann, F.; Neugebauer, J.; Raabe, D.: Phys. Rev. Mater. 4 (2020) 033601.

76. Rao, Z.; Dutta, B.; Körmann, F.; Lu,W.; Zhou, X.; Liu,C.; Kwiatkowski da Silva, A.; Wiedwald, U.; Spasova, M.; Farle, M.; Ponge, D.; Gault, B.; Neugebauer, J.; Raabe, D.; Li, Z.: Adv. Funct. Mater. 31 (2021) 2007668.

77. Friak, M., Bursikova, V., Pizurova, N., Pavlu, J., Jiraskova, Y., Homola, V., Mihalikova, I., Slavik, A., Holec, D., Vsianska, M., Koutna, N., Fikar, J., Janickovic, D., Sob, M., Neugebauer, J., Crystals 9 (2019) 299.

78. Rao, Z.Y., Ponge, D., Körmann, F., Ikeda, Y., Schneeweiss, O., Friak, M., Neugebauer, J., Raabe, D., Li, Z.M., Intermetal. 111 (2019) 106520.

79. Yang, D.C., Jo, Y.H., Ikeda, Y., Körmann, F., Sohn, S.S., J. Mater. Sci. Tech. 90 (2021) 159

80. Guo, W., Su, J., Lu, W., Liebscher, C.H., Kirchlechner, C., Ikeda, Y., Körmann, F., Liu, X., Xue, Y., Dehm, G., Acta Mater. 185 (2020) 45.

81. Kies, F., Ikeda, Y., Ewald, S., Schleifenbaum, J.H., Hallstedt, B., Körmann, F., Haase, C., Scripta Mater. 178 (2020) 366.

82. Rogal, L., Ikeda, Y., Lai, M., Körmann, F., Kalinowska, A., Grabowski, B., Mater. Design 192 (2020) 108716.

83. Rao, Z., Dutta, B., Körmann, F., Ponge, D., Li, L., He, J., Stephenson, L.T., Schäfer, L., Skokov, K.P., Gutfleisch, O., Raabe, D., Li, Z., Phys. Rev. Mater. 4 (2020) 014402.

84. The SPHInX multiscale program library is available at http://sxrepo.mpie.de. See also Boeck, S.; Freysoldt, C.; Dick, A.; Ismer, L.; Neugebauer, J.: Comp. Phys. Commun. 182 (2011) 543.

85. http://potentials.rub.de/

86. https://pyiron.org/phasediagram-workshop-2020/Introduction.html

87. Kirchmayer, A., Lyu, H., Pröbstle, M., Houllé, F., Förner, A., Huenert, D., Göken, M., Felfer, P.J., Bitzek, E., Neumeier, S.: Adv. Engin. Mater. 22 (2020) 2000149.

Department of Interface Chemistry and Surface Engineering (GO)

M. Rohwerder[1], J. Neugebauer[2] (provisional department head)

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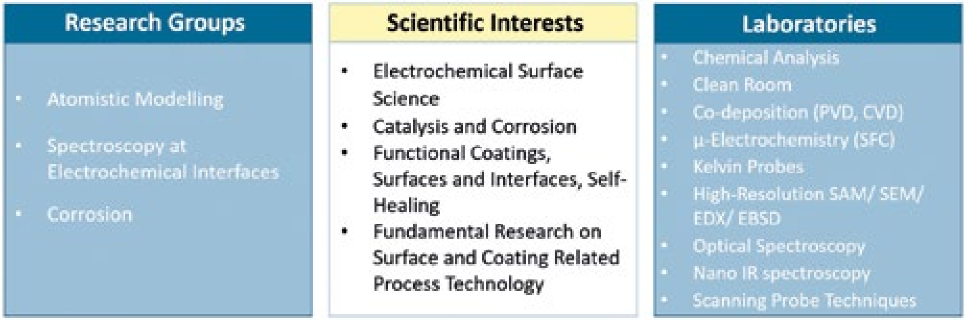
Introduction

The Department of Interface Chemistry and Surface Engineering (GO) is mainly focussing on corrosion and electrochemical energy conversion. It is internationally known to be one of the leading groups in the field of electrochemical sciences. Our mission is to combine both fundamental and applied sciences to tackle key-questions for a progress towards new or better, energy saving and efficient, cheaper and longer lasting materials for applications as structural (in particular steels and other alloys) and functional materials, e.g. for fuel cell catalysts, pre-treatments and smart coatings amongst others. The department currently hosts four research groups. The different groups focus on high-throughput methods and the development of combinatorial methods in adhesion science and electrochemistry, on characterization of electrified interfaces by complementary methods such as vibrational spectroscopy, *in situ* diffraction studies and scanning probe techniques, such as Scanning Flow Cell (SFC), Scanning Kelvin Probe (SKP), Atomic Force Microscopy (AFM) or Scanning Tunnelling Microscopy (STM), as well as on related *ab initio* simulation. We combine electrochemistry with a surface and interface science approach, and in most projects we complement both, experimental studies on atomically well-defined model systems as well as on technical systems with atomistic *ab initio* modelling. All groups in the department have their independent and strong research agenda, while collaborative research projects are synergistic and focus on major challenges and complex scientific questions that require the scale and interdisciplinarity.

Of crucial importance in the last three years was to stabilize the department. Since the head of the department M. Stratmann, took over as president of the Max Planck Society in June 2014 and is officially on leave from his position at the MPIE, significant changes took place in the department. M. Rohwerder took over the coordination of the department and J. Neugebauer (head of the department "Computational Materials Design") acts as temporary head of the department. Most notably the number of research groups shrank quickly from six in 2014 to two in 2019. However, the research group of A. Erbe (Optical Spectroscopy) remained very active as a guest group until end of 2019, and the one of K. Mayrhofer (Electrocatalysis) too, to be followed by the very active guest group of O. Kasian (Dynamic Electrocatalytic Interfaces, until end of 2021). In 2020 the early career research group "Spectroscopy at Electrochemical Interfaces" was established in the GO department as joint research group of the MPIE and the cluster of excellence RESOLV (www.solvation.de) located at the Ruhr-Universität Bochum (RUB). The group uses interface spectroscopic methods to study solvation and other dynamic effects on solid surfaces interfaces (see p. 45). By establishing this new group important expertise could be regained.

The department is actively participating in various collaborative research efforts with internal and external partners. An important example is the German Research Foundation (DFG) Cluster of Excellence "Ruhr Explores Solvation" (RESOLV), which is centred at the RUB's Faculty of Chemistry and Biochemistry. Our contributions to solvation science in RESOLV focus on the role of solvents in electrochemical reactions, and solvation of interfaces (see p. 86).

With its versatile experimental and theoretical expertise (Fig. 1) the GO department is in the unique position to assist and promote also diverse **industrial research especially in investigating the underlying fundamental electrochemical reaction mechanisms and kinetics**.



Research Groups

- Atomistic Modelling
- Spectroscopy at Electrochemical Interfaces
- Corrosion

Scientific Interests

- Electrochemical Surface Science
- Catalysis and Corrosion
- Functional Coatings, Surfaces and Interfaces, Self-Healing
- Fundamental Research on Surface and Coating Related Process Technology

Laboratories

- Chemical Analysis
- Clean Room
- Co-deposition (PVD, CVD)
- µ-Electrochemistry (SFC)
- Kelvin Probes
- High-Resolution SAM/ SEM/ EDX/ EBSD
- Optical Spectroscopy
- Nano IR spectroscopy
- Scanning Probe Techniques

Fig. 1: *Current research portfolio and expertise.*

For instance, the electrochemical oxygen reduction reaction (ORR) - a key process in corrosion as well as energy conversion - has been the focus of several joint research projects in the department aiming at a fundamental understanding of its mechanism for better control in corrosion protection as well as improved catalysis in fuel cells and air batteries. These research projects combine both advanced electrochemical as well as *in situ* and in operando spectroscopic experiments. In the reporting period besides ORR also the oxygen evolution reaction (OER) has become object of intense research activities and in this context also research on transpassivity of electrode materials under OER conditions (see e.g. “Electrocatalysis” and “Spectroscopy at Electrochemical Interfaces” reports below). The knowledge obtained in the fundamental research on electrochemical key reactions such as oxygen reduction and oxygen evolution play a crucial role in helping us to unravel practical problems met in industrial applications or processes such as in corrosion, batteries and fuel cells.

Several research projects focus on industrial processes such as pickling and alternative surface treatments. A special collaborative interest of the institute is hydrogen in steel. The activities in this field have been further enhanced, complementing experimental and theoretical methods in cooperation between the departments play an important role. In the GO department in particular the Scanning Kelvin Probe and Scanning Kelvin Probe Force Microscopy were further developed in combination with complementing techniques to uniquely and directly detect hydrogen in steel and even through zinc-based alloy coatings.

Fig. 2: *Part of the GO department with M. Stratman during an excursion at the department retreat at Ringberg Castle in 2021.*

Scientific Concepts and Broader Impact

All our research groups are synergistically working on common areas of interest and we are strongly dedicated to collaborative work with external partners. Our scientific concept comprises diverse fundamental and industrial fields:

Electrochemical sciences

Fundamental research on electrochemical interfaces is a key activity within the GO department as an in-depth characterisation of these interfaces or interphases is of indispensable importance for a full mechanistic description of electrochemical processes in the key areas corrosion and electrocatalysis. In the recent years extensive collaborative experimental and theoretical work focussed especially on the electrochemical oxygen reduction reaction (ORR), a fundamental electrochemical reaction relevant in both corrosion as well as energy conversion processes. At metal surfaces the ORR is the cathodic partial reaction driving many corrosion processes. On the other hand, the electrocatalysis of the ORR in fuel cells and air batteries is intensely studied with the aim of improving the kinetics and reducing the over-potential that limits the energy efficiency. One important approach for advancing our understanding of this reaction is to make progress in according *ab initio* simulations. Comparison with experimental results, however, is difficult, because reliable experimental *in situ* data for instance of reaction products are difficult to obtain from the electrochemical double layer region, which is buried from most surface analytical techniques by a layer of bulk electrolyte. Thus, experimental feedback is more or less missing. Of increasing importance here is our surface science approach for fundamental investigation of electrode surfaces. This is a long-term research topic within the department. For this we have developed a novel methodology that allows to fully control not only the potential of electrodes covered by just ultrathin electrolyte layers, but also to measure the full current density-potential correlation (I(U) curves) for electrochemical reactions such as oxygen reduction on that same electrode. This methodology is based on combining Kelvin probe with a hydrogen permeation experiment, which allows for the first- time direct measurement of e.g. the oxygen reduction kinetics at the buried interface, first performed and proven to work in a modified Devanathan-Stachurski set-up where at the exit side the potential was measured instead an oxidation current density. These electrodes are just covered by ultrathin electrolyte layers denoted by us as “electrodes in the dry”. The humidity of the environment can be adjusted to different partial pressures of water, thus adjusting the exact thickness of the electrolyte layers. This novel approach enables us to control the structure of the double layer in a so far unimaginable way. For instance, under full potential control electrodes with double layers containing water in the sub-monolayer range can be realized and still the full current-potential dependence for electrochemical reactions such as oxygen reduction can be obtained. For the first time this should make a fully controllable electrode accessible for analytical techniques for in operando investigation without any restrictions, as for instance surface analytical tools such as near ambient pressure x-ray photoelectron spectroscopy (NAP-XPS) and infrared spectroscopy. In accordance with the importance of the oxygen reduction reaction for the department, the main activities of the current research focus on this reaction. However, most of this work so far has been carried out on palladium terminated surfaces. Hence, besides investigating ORR on such electrodes, also very fundamental research on the general applicability of the "electrode in the dry" concept on different electrode materials, such as Pt, Ir and Au, is carried out (see "Corrosion" group report below).

This experimental part of our activities on electrochemical sciences is accompanied by work in the atomistic modelling group where in cooperation with the CM department a novel approach to control the electrode potential was developed, that can be used equally well to perform empirical or *ab initio* molecular dynamics simulations (see "Atomistic Modelling" group report).

Many of the research projects allocated in the field of electrochemical sciences are closely related to the DFG-funded Cluster of Excellence RESOLV with the RUB. One example is the "electrodes in the dry", but the main activities are carried out in the MPIE-RUB early career research group "Spectroscopy at Electrochemical Interfaces" (see below and p. 45).

Corrosion

The aim of the department is to obtain fundamental insight into corrosion mechanisms with the aim to develop advanced countermeasures.

During the last three years the corrosion research of the department covered a wide range of topics in the areas of aqueous and atmospheric corrosion processes and their inhibition, covering both fundamental and applied aspects. A key technique for corrosion research remains the Scanning Flow Cell (SFC). Using the SFC system with downstream analytics such as mass spectroscopy or UV-vis analysis allows electrochemical high-throughput screening and characterization of corrosion properties of material samples. This method, which was developed in the GO department, has become a routine technique for corrosion research and was applied on a wide range of materials, also in the last three years.

The investigations by SFC are mainly focused on the fundamental active dissolution kinetics. Atmosphere change experiments in the Kelvin Probe, on the other hand, are providing information primarily about the reactivity of the surface oxides of the as prepared samples and to some extent the initial stages of long-term performance, depending on the exact exposure conditions and the duration of the experiments. One example is the performance of zinc alloy coatings. The effect of changes from oxygen free to oxygen containing atmospheres and back on the potential measured by Kelvin probe on the surface of such a coating are taken as an indicator for the reactivity of the corresponding surface oxide, which was indeed found to correlate well with observed corrosion behaviour. However, it was now found that there are also exceptions from this (see "Corrosion" group report). Concerning atmospheric corrosion, our main focus is on the delamination of organic coatings. Organic coatings are commonly employed to protect materials against corrosion. On steel, including galvanised steel, cathodic delamination is the main mechanism of failure of these coatings. In this delamination mode oxygen reduction at the buried interface and especially the radicals produced as intermediates or side products play a crucial role in destroying the adhesion at the interface. In order to improve the delamination resistance, the standard strategy is to use pre-treatments prior to application of the organic coating that effectively inhibit electron transfer reactions at the interface and thus also oxygen reduction. Examples are chromatation and phosphatation, where the first is more or less fully banned now and on the latter there is strong pressure to replace or even skip it. GO department is involved in corresponding research. The main problem, however, is that any development of novel pre-treatments and coating concept is slowed down by the required long-term evaluations. Unfortunately, up to now no real break-through has been made in a simulation of the delamination process and long-term prognosis. An important requirement for such simulation is of course a deep insight into the underlying mechanisms and knowledge about the key processes. In the reporting period important insights were obtained on the fundamental mechanisms of this process (see p. 85 and below).

High temperature reactions

Reactions at high temperatures is another topic that is investigated within the GO department. Special focus has been on short term high temperature treatments that are crucial in many industrial processing steps in steel making, such as hot rolling and recrystallization annealing before hot dip galvanizing, where they cause significant external and internal oxidation. These relatively short processing steps are generally not much investigated yet and are determined by kinetics of oxygen uptake, nucleation and growth of oxides and only to limited extent by diffusion, usually considered of key importance in high temperature oxidation, which leads to final morphologies that are often far from equilibrium. More recently this research is replaced by investigations on direct reduction of iron ores by hydrogen (see "Corrosion" group report).

Functional surfaces, interfaces, coatings and materials

Our research on functional surfaces, interfaces and coatings is mainly focused on improving the long-term stability of coatings applied on metallic substrates. The targeted main functionalities of coatings and interfaces are smart sensing of and protection against corrosion. For this, new concepts are developed and in-depth investigation of crucial fundamental processes were performed. Of especial importance for achieving high-performing self-healing response are fast trigger signal spreading and also sufficiently high transport of active agents from within the coating to the defect site. This requires an in-depth fundamental understanding of these processes and about the possibilities to optimize them. The related research covers the whole coating system, from nano-containers for storage of active agents, over suitable active agents to tailoring properties of all involved surfaces and interfaces. Especially promising for optimized trigger signal spreading and transport of active agents seem to be interfacial layers of conducting polymer (see “Corrosion” group report).

Also, bulk functional materials are investigated, such as e.g. quantum dot solids which are of high interest for creating novel quantum materials with targeted properties with possible applications, e.g., as next-generation light absorbers for photovoltaics, photoelectrochemistry and power electronics. III-V semiconductor-based Quantum dot solids, consisting of e.g. InP or InGaP nanocrystals, are among the most widely investigated materials systems due to their potentially superior optical properties and lower toxicity, compared to commercially available II-VI quantum dot solids. Whereas narrow emission line widths are readily achieved in II-VI quantum dot solids, III-V systems still suffer from inferior colour purity due to broad emissivity and poor photoluminescence quantum yields. We recently demonstrated that nanocrystal stoichiometry dispersion is a major source of trap states and largely responsible for the observed emission broadening (see “Atomistic Modelling” group report).

Another important topic are electro responsive ‘smart’ interfaces which are of interest for applications in microfluidics, separation systems, biosensors and -analytics. (see “Spectroscopy at Electrochemical Interface” group report).

Industrial processes

The expertise of the department is of great relevance for the investigation of fundamental aspects of industrial processes.

Fundamental problems of oxidation and hydrogen uptake during industrial production steps of high strength steel sheet have been a longstanding focus of the department and many of the activities within the reporting period on these topics are based on that earlier work. A high performing high temperature lab is available for fundamental investigations of according industrial processes, which is currently restructured in order to support the new inter-departmental focus on direct iron ore reduction.

Joining different materials by welding is an important industrial process, but some material combinations are not accessible in the classic welding processes that involve melting. In collaboration with the Institute of Production Engineering and Forming Machines of TU Darmstadt the role of surface treatments on the interface chemistry on cold welding of steels and aluminium alloys was investigated (see below reports by the “Corrosion” group and the former “Interface spectroscopy” group). The department was here mainly involved with chemical and electrochemical surface treatment and *ex situ* investigation of the resulting interface.

Scientific Groups

Corrosion (M. Rohwerder)

The main scope of this group is to address fundamental questions of corrosion and surface and coating technology by isolating the crucial problems behind them and designing model experiments and model samples for their systematic investigation. Three exemplary research projects, in collaboration with external partners, that stand quite nicely for the research in the group are: “H2 free - Investigation and modelling of hydrogen effusion in electrochemically plated ultra-high-strength-steels used for landing gear structures” within the CleanSky programme of the Horizon 2020 - Research and Innovation Framework Programme; ”MAXCoat - MAX-Phase coatings for corrosion protection of bipolar plates for mobile fuel cells”, financed by the Federal Ministry for Economy and Energy; and “PredictCorr - Prediction of durability and lifetime of organic coated metals under long-term environmental conditions”, financed by the Flamish Research Foundation (see p. 85).

In detail the main activities on the research interests in the Corrosion group can be summarized as follows:

1. Elementary steps of electrochemically driven de-adhesion of organic coatings

Fundamental research on coating delamination is of paramount interest for the department. This is, however, very challenging, because coating delamination is extremely complex and the buried interface is difficult to investigate, as it is inaccessible for most analytical tools. A huge breakthrough was the new approach based on a potentiometric measurement of the equilibrium potential of the oxygen reduction and oxidation of hydrogen permeating from the backside of the sample to the buried interfaces, denoted by us as permeation based potentiometric method (ppm). This approach overcomes the main obstacle preventing electrochemical measurements at the buried interface, the high resistance of the organic coating against ionic current that makes a controllable polarization of the interface by standard three electrode set-up impossible. From the hydrogen uptake on the entry side thus a full current-potential relationship curve (I (U)) can be constructed, as could now be shown even for the case that the exit side is not immersed into electrolyte [1], just as it is of relevance for the situation met during corrosion driven delamination under atmospheric condition.

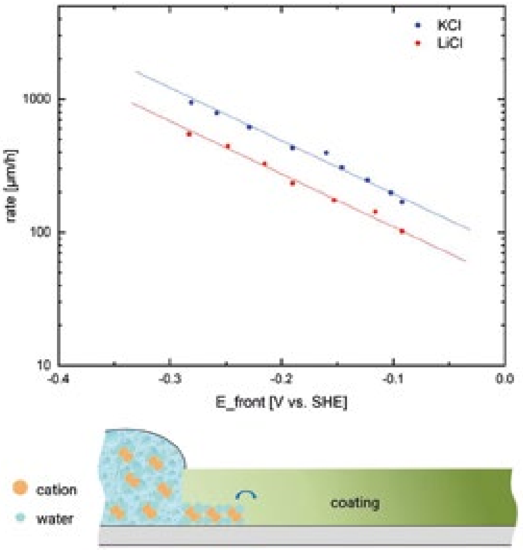


Fig. 3: *Top: delamination rates measured for a PVB (polyvinlybutyral) coating applied onto iron, for either KCL or LiCl as electrolyte in the defect, plotted as a function of the potential at the delamination front. Since the rate also depends on cation size, oxygen reduction seems not the rate determining step here. It is assumed that rather the insertion of cations into the yet intact interface (bottom) is the rate determining step.*

The investigations focus on two sets of samples: one are well defined samples based on inert noble metal, which allow well controllable structuring of the buried interface and integration of functional groups e.g. by use of self-assembled molecular films at the interface [2-4], which are considered for developing dedicated model samples for very fundamental studies. The other set is currently more important and is focussed on technically more relevant samples, such as coated iron/steel and zinc, and some experiments were also performed on coated aluminium alloy. In the reporting period the main focus was on coated iron and steel, mainly within the PredictCorr project (see p.85). The idea of that project is to provide the tools for a long-term prediction of coating performance. At the MPIE the focus is on unravelling the underlying mechanisms and providing information about the corresponding kinetics. One planned activity is to investigate the oxygen reduction kinetics at the buried interface between metal and organic coatings, using the hydrogen permeation based method developed in the group [1]. To be precise, the investigations should include the interface at different stages of the delamination process: the intact interface, where degradation is initiated right at the delamination front, in the delaminating area, where there will be furthermore different stages of delamination, depending on the exact position, and within the delaminated area. Hence, owing to the complexity of this task it was decided to focus first on the very first step of delamination, i.e. is the insertion of cations, migrating from the defect site, into the intact interface. This leads to a correlated decrease in potential at the interface and thus enabling the onset of oxygen reduction. Hence, this can be considered as the first step of delamination. So far, the role of cations in cathodic delamination was solely discussed as migration of cations from the corroding defect to the intact interface, i.e. along the delaminated interface. This cation migration is required to ensure charge neutrality, i.e. to compensate the electron flow along the interface from the defect to the delaminating and delaminated interface, that is required for the oxygen reduction reaction at the decreased potentials. It was assumed until now, based on the work by M. Stratmann et al. that a square root dependence on time for the overall delamination rate is associated with cation migration as a rate determining step. A linear time dependence, on the other hand, is generally believed to be indicative for oxygen reduction as rate determining step. However, as mentioned, the very first step has to be that cations move into the intact interface, which, if rate determining, is expected to also possibly lead to a constant delamination rate. The idea for investigating this in more detail was at first to perform studies in the absence of oxygen, such as e.g. in humid nitrogen. In that case no oxygen reduction occurs and hence no delamination, i.e. the interface stays undamaged. It was already shown by M. Stratmann et al. that in that case the ingress of cations into the intact coating can be monitored by Scanning Kelvin Probe (SKP). It seems reasonable to assume that the overall rate of this cation ingress is determined either by the migration of cations along this interface or by the first insertion of cations in the yet cation free interface at the migration front. Within the PredictCorr project different model samples, featuring different coating types and different surface treatments of the underlying steel were provided and it was found that for most of these the overall rate was depending on the square root of time and that the initial migration behaviour in nitrogen atmosphere was nearly identical to the one of delamination. This is difficult to explain, if cation migration was the rate determining step of delamination, as in the one case the cations migrate along a delaminated interface, in the other case along an undamaged, intact one (see p. 85). Hence, it seems likely that the rate determining step in both cases might be the initial ingress of cations into the yet unaffected interface, also denominated in the group as cation insertion. Also, other indications for this were discovered in other research projects. For instance, it was found that adsorbed species such as CO_2 [5] on coated zinc and even O_2 on novel chromium coatings [6] have a huge effect on delamination/migration. Since during delamination the delaminated interface is characterized by relatively high rates of oxygen reduction and for the case e.g. of zinc also by anodic zinc oxidation and dissolution, it seems unlikely that the presence of small amounts of CO_2 could have a significant effect on ion mobility at that interface. This means that the observed effects are most likely rather related to the intact interface, i.e. the initial cation insertion step into the intact interface.

Related observations were made within PredictCorr, too. For instance, the observed initial similarity between delamination behaviour in air and migration behaviour in nitrogen was found to change after a while (see p. 85).

For some of the coating systems the migration in nitrogen atmosphere slowed down after a few hours and in some cases even came to a total halt. It is assumed that this is due to changes at the interface caused by the change of atmosphere and is object of intense current research. Despite of the unexpected high complexity also of the cation insertion and migration processes, it was possible to make significant progress. Key to this was a new experimental procedure wherein the defect edge, where the delamination is initiated, is not left to freely corrode, but instead is stepwise polarized to a sequence of well controlled potentials. Thus, it was found that the cation insertion process at the delamination front depends sensitively on electrode potential at the front, but also on cation size (Fig.3). This is object of intense current research.

2. Intelligent self-healing concepts for corrosion protection

In the last three years the research on intelligent self-healing concepts for corrosion protection could build on many years of intense activities on that topic. While in prior works especially safe storage of active agents and their corrosion triggered release from capsules were at the focus of our research, as well as the functionality of intrinsically conductive polymers (ICP) as switchable encapsulation material and for use as isolated agglomerates for reducing delamination rate by surrounding protection zone effects [7, 8], the focus of the activities has now shifted towards additional coating functionalities, such as corrosion sensing and signalling within coatings [9], as well as combinations [10] , or improving the transport of active agents for corrosion inhibiting and/or restorage of damaged organic coating from their storage site inside a coating towards the defect. For the latter once more ICPs were investigated. While in the last reporting period it could be shown that ICP films can significantly enhance trigger signal spreading, thus ensuring a fast and widespread release of active agents upon onset of corrosion, now it was investigated in how far they also can enhance the transport of the released active agents towards the defect site. For this, a dedicated novel experimental set-up was designed and successfully utilized (Fig.4) and it could be shown that indeed especially for cationic and neutral active agents a significantly enhanced supply of these can be provided by large networks of (partially reduced) ICPs [11].

The reason for the excellent cation mobility in the partially reduced ICP network is that by electrochemical reduction ICPs are becoming at least to some extent cation-permselective. It is assumed that the also observed very high mobility of the uncharged active agents might be due to a correlated switch from rather hydrophobic to rather hydrophilic behaviour of the ICP upon (partial) reduction. This is object of current research.

Hence, in sum it seems that contrary to longstanding assumptions, based on our earlier findings, that large networks of ICP, such as continuous ICP films or extended agglomerates of ICP particles, would rather reduce the corrosion protection properties of a coating than improve it, when making use of these new functions, such extended networks might allow realizing novel smart corrosion protection coatings with significantly improved self-healing capabilities.

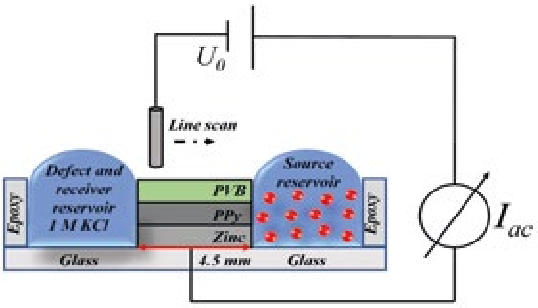


Fig. 4: *Schematic drawing of the set-up used for the transport studies: the coating system is placed between two reservoirs. One is for simulating the defect (left) and the other serves as reservoir for active agents. This allows to study the effect of variations in the coating system and of the nature of the different active agents on their transport through the bulk coating and alongside the different interfaces [11].*

However, for this it is important to ensure that the spreading of ICP reduction from a corroding defect site is at some point stopped, even if the corrosion in the defect cannot be stopped and continues. Otherwise, the coating would quickly fail over a large area. Now it was found that by use of suitable counter-anions, the reduction and delamination of continuous ICP layers cannot only be stopped after a certain while, but both can be reversed, i.e. a full self-healing of the ICP layer and restorage of the interface can be achieved. This is an important requirement also for full self-healing: the ICP film is quickly reduced and the interface delaminated for fast signal spreading, release and transport of active agents. But upon healing of the defect both ICP film as well as interface are restored again. Importantly, this occurs not only readily when the defect is successfully inhibited, but also without that, preventing disastrous failure of the coating. How that works even in the presence of a still actively corroding defect is, however, not fully understood yet, but object of intense current research. A new activity within the department is research on novel inhibitor concepts for mitigating delamination and corrosion, see e.g. [12].

3. Corrosion

While in the past the main corrosion activity within the department was mainly on organic coating delamination and the role of the metallic substrate (or rather the passive layer on its surface) beneath the organic coatings, in the last three years more classical corrosion topics have re-gained importance. Most notably passive layers and their failure are now of increasing research interest in the department, see e.g. [13, 14]. One main motivation for this is the search for novel materials for bipolar plates that are used in fuel cells. The challenge here is that on the one hand a highly passive surface layer is targeted, on the other hand at the same time a high conductivity. This is object of current research within the project MaxCoat (in cooperation with the Zentrum für Brennstoffzellentechnik in Duisburg and the Leibniz-Institut für Plasmaforschung und Technologie in Greifswald), where MAX phases, which are known to show high conductivity and high chemical stability, are investigated as a possible coating material for bipolar plates. Fast feedback on even smallest defects and the general electrochemical performance of these coatings is provided at the MPIE by means of the scanning flow cell method in combination with inductively coupled mass spectroscopy (SFC-ICPMS). This approach was also successfully applied in other projects, such as e.g. elucidating the corrosion protection performance of magnetron sputtered TiMgN hard coatings [15].

In fuel cells potential-triggered passivation of e.g. TiC supports may have a great potential for increasing lifetime of Pt-catalysts. Our results have shown that the deposition of a closed Pt film onto a TiC support protects the latter from dissolution up to about 1.05 VRHE. At higher potentials where Pt oxidation and dissolution occur, the underlying TiC heals out the defects and imperfections created in the Pt film by building up a protective oxide layer that stops further degradation [16].

Also, corrosion properties of novel light-weight materials are investigated. For instance, in cooperation with H. Springer from the MA department the effect of Al and Cr additions on the mechanical, physical and corrosion properties of Fe was investigated in a combinatorial approach for screening suitable compositions as the basis for the alloy design of novel lightweight corrosion resistant steels. It was found that upon decrease of Cr content in the alloy and increased Al content, also in the passive layer chromium is more or less replaced by aluminium while a quite significant degree of passivity is preserved [14].

On the other side of the spectrum of our activities on corrosion were studies on erosion-corrosion, with a focus on degradation processes as they occur in pipelines transporting slurries e.g. in the mining industry [17], and investigations on the effect of surface treatments on the stability and degradation of cold pressure welded components [18, 19].

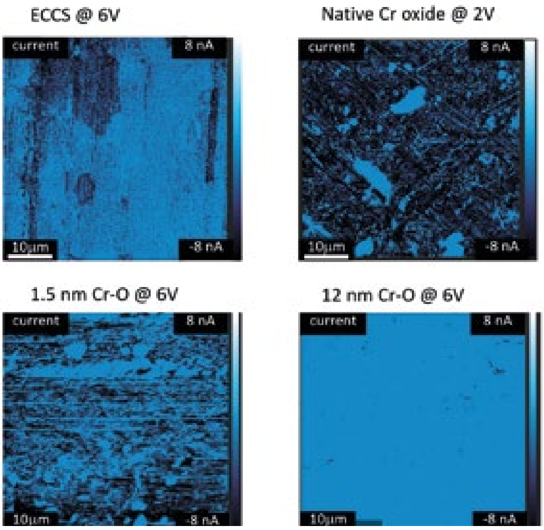


Fig. 5: *Current maps obtained by current sensing atomic force microscopy (CS-AFM) on differently terminated chromium layers (note the lower applied bias between tip and sample for the native chromium oxide covered sample). ECCS denotes the standard chromium coating deposited electrolytically from Cr(VI) containing electrolyte, which has to be replaced for environmental and safety reasons, and Cr-O, an electrochemically deposited additional chromium oxide film on top of a chromium layer deposited from trivalent Cr containing electrolyte (1.5 and 12 stand for 1.5 and 12 nm of the Cr-O film). The differences between the different samples are of interest as well as the heterogeneity.*

4. Semiconducting properties of surface oxide films

The semiconducting properties of surface oxides play a crucial role especially in atmospheric corrosion and coating delamination. One huge research field are zinc alloy coatings, where alloying with magnesium and aluminium can result in significantly enhanced performance. In general, the composition, structure and the related properties of oxides are of utmost importance for the performance of coating systems applied on metals. In the reporting time an important activity was the investigation of the oxide layers on electrochemically deposited chromium coatings on steel, where strong differences in conductivity were found for different kinds of the terminating chromium oxide layers (Fig. 5).

Especially insulating was a strongly O-deficient metastable Cr-O phase [6], where already ultra-thin films showed only extremely low conductivity. Also, cathodic delamination was found to be strongly inhibited by such oxide films, which at the first look would be assumed to be directly linked to the low conductivity which would also suppress oxygen reduction.

However, it was also found that cation mobility on these oxides is also extremely low and interestingly depends on partial pressure of oxygen in the environment. By ambient pressure photo-electron spectroscopy (AP-XPS) it was found that indeed oxygen adsorption on these oxides is sensitively responding even on slight changes of oxygen partial pressure. This oxygen adsorption results in quite significant changes in potential upon switching between nitrogen and oxygen containing atmospheres, as can be measured by SKP. This has never been observed before for such delamination resistant samples. Usually a large response is a sign for fast delamination behaviour.

More recent is our research on the effect of hydrogen on the properties of the oxide in the passive layers of metals. Our motivation for this research is to investigate how the work function of different oxide, or rather the electrode potentials established on their surface in different environments, depends on hydrogen activity. This knowledge can be used e.g. for the study of oxygen reduction at buried interfaces, and for hydrogen detection in or measurement of its permeation through materials at high local resolution. This is closely related to our research on the “Electrochemistry in the “dry”.

5. Electrochemistry in the “dry”

Electrodes covered by just ultra-thin electrolyte layers are another important topic within the corrosion group. This is of practical importance e.g. for our fundamental studies on atmospheric corrosion, where we take the potential change on the (oxide covered) surface of a metal upon switching the atmosphere between air and nitrogen and back as an indicator for its reactivity. However, electrodes in the “dry” are of general importance. Many phenomena in corrosion and electrochemistry are occurring on electrodes that are not immersed into bulk electrolyte or covered by bulk-like electrolyte layers, and these are still not well understood. Maybe the most fascinating result of this work is that the electrochemical oxygen reduction reaction on the surface of palladium just covered by one to two monolayers of water, is in principle the same as on an electrode immersed in acidic bulk electrolyte, just without the mass transport limitation for oxygen [1]. Because for an electrode immersed in electrolyte the double layer is more extended, this directly raises questions about the structure of the electrochemical double layer, which is here confined to just one or two monolayers with protons as counter ions, and how this determines the reaction kinetics. This question is at the focus of intense research on emersed electrodes, where it is possible to include also other ions into the electrochemical double layer and to tune the water layer thickness via the relative humidity in the atmosphere the emersed electrode is exposed to. It could be shown that during emersion even into dry nitrogen atmosphere a water layer is preserved and that the water molecules in this layer are assuming an orientation that is determined by the potential. The effect of additional water layers, adjusted by higher relative humidity, is object of current research, as well as the question what their effect is on the kinetics of reactions such as oxygen reduction. While the investigations on emersed electrodes are so far mainly carried out for gold as electrode material, the “hydrogen electrode in the dry” concept is investigated in parallel for other materials besides palladium [1], such as iridium and gold. The idea is to combine the concept of the emersed electrode with the concept of polarisation from the backside by adjusting there a well-defined H activity [1], usually by electrochemical polarisation. Interestingly, the “hydrogen electrode in the dry” on Ir and Au is depending sensitively on relative humidity. While for Pd there is 1:1 relation between potential applied from the backside and potential measured by Kelvin Probe on the “dry” electrode in low as well as in high relative humidity, for Ir, for instance, such a 1:1 correlation is observed only at high humidity, while at lower humidity the response is less than 1:1, the closer to it the higher the humidity. This is object of current research. Interestingly, the linear relation remains. It should be pointed out here, that it still remains unclear why a linear relationship is observed in the first place, since as the equilibrium reaction on the electrode surface is: $H_{ab} \leftrightarrow H^{ad} \leftrightarrow H^{+}+e^{-}$. Hence, an increase in H in the metal should lead to an increase in H^{+} in the nanoscopic layer, because the ultra-thin water layer in contrast to bulk electrolyte has no buffering capability. However, the observed behaviour, i.e. the linear 1:1 correlation between applied and measured potential, is in accordance only with a pinned pH. First results obtained by in operando study of such a hydrogen electrode on palladium indicate that adsorbed CO_2 contamination might play a role in the pH buffering. On iridium, however, significantly lower contamination levels have been observed, but still a linear correlation is found. The role of contamination layers is one aspect to be studied especially with ambient pressure photoelectron spectroscopy (AP-XPS). Also, for the planned combination of emersed and dry electrode AP-XPS will play a crucial role, but a new experimental AP-XPS set-up had to be designed for that and is currently being built up. This research on electrodes in the “dry” will also play a role in the GO department part within the continuation of the excellence cluster RESOLV.

Besides this very fundamental focus on the electrochemical double layer, also more applied aspects of the electrode in the “dry” are investigated. One is to use the changes in potential induced upon switching from humid nitrogen to hydrogen containing atmospheres on the surface of a metal for obtaining information on correlated adsorption energies at the corresponding solid/liquid interface and for providing insight into their role in electrocatalysis of the hydrogen evolution reaction [20, 21]. Another important reaction is of course oxygen reduction, which is a key reaction e.g. in corrosion, coating delamination as well as in fuel cells. Our research could provide very strong indications that the “bare” catalyst particles, which are just covered by adsorbed water in the monolayer range, can be electrochemically active and can provide ORR at high rates. For ORR on a “bare” catalyst particle in a membrane electrode assembly (MEA), protons have to be delivered from the ionomer in the vicinity. It is reported that proton transport can readily occur on the surface of the metallic catalyst, e. g. for Pt via Pt-OH or Pt H. Thus, as supported by our results, catalysts not covered by ionomer may play an important role in the overall ORR activity of the MEA. [1].

6. Measurements of Hydrogen distribution and effect on embrittlement

The novel SKP and SKPFM based method for hydrogen mapping by making use of the "hydrogen electrode in the dry" has by now become an established tool for our research on hydrogen related topics, in close cooperation with other departments [22, 23].

Trying to optimize how to carry out high resolution mapping of local hydrogen distribution also of materials with very fine microstructure is a key focus of current activities. This is made difficult by too high back-ground intensities of hydrogen and is tried to be solved by preparing much thinner samples and/or restricting hydrogen loading to an ultra-thin zone at the surface, as far as that is feasible (see p. 68). Especially noteworthy is another also quite fundamental research project where we try to evaluate the importance of the full 3D-parameter range of mechanical strain, hydrogen activity and exposure time on hydrogen induced material failure. This interesting project was unfortunately especially severely delayed by the pandemic, but is now going into a very promising direction.

In another research project (in the Horizon 2020 framework (H2Free)) it is tried to obtain important input data for the modelling of hydrogen release from zinc-alloy and cadmium coated landing gear components. This is a very challenging task and a combination of novel cutting-edge application of Kelvin probe for measuring hydrogen permeation and its distribution inside the materials and thermo desorption analysis is applied. It was found that deformation layers at the surface of the high strength steel are significantly complicating the analysis, leading to a very challenging task.

7. High temperature reactions: from grain boundary reduction to direct reduction of iron ores by hydrogen and ammonia

In cooperation with the Universität Duisburg-Essen and the University of Waterloo also graphene deposition on GaN was investigated. One of the bottlenecks in the implementation of graphene as a transparent electrode in modern opto-electronic devices is the need for complicated and damaging transfer processes of high-quality graphene sheets onto the desired target substrates, such as GaN. By replacing the commonly used hydrogen (H_2) process gas with nitrogen (N_2), it was possible to suppress GaN surface decomposition while simultaneously enabling graphene deposition at < 800 °C in a single-step growth process. At the MPIE mainly characterization via XPS was carried out, demonstrating that the result exceeds the so far best reported values for directly grown graphene on GaN in literature [24].

The investigation of high temperature reactions has been a key expertise within the department for decades. While in the past mainly oxidation processes were investigated in our high-temperature lab, such as in the reporting period the selective oxidation at the grain boundaries of various binary and ternary iron-based alloys [25, 26], the focus is now mainly on investigating the direct reduction of iron ores by hydrogen or ammonia.

Our research on internal oxidation of Fe-Mn-Si alloys [25, 26], which addresses the problem of grain boundary oxidation upon hot rolling of steels, revealed that the extent of the oxidation increases significantly with increasing Si content. Interestingly, an external $(FeO)_{1-x}(MnO)_x$ layer of several tens of nanometres thickness was formed on all the alloys. Nevertheless, the external layer did not impose diffusion inhibition on the oxygen uptake, but this $(FeO)_{1-x}(MnO)_x$ layer was found to have a crucial influence on the kinetics at the studied annealing conditions. As the FeO content of the oxide layer increases with more silicon concentration in the alloy, the layer is suggested to facilitate the O-uptake kinetics [25]. In more fundamental studies on pure iron and binary Fe-Si and Fe-Mn alloys, carried out in our ambient pressure XPS set-up, it was found at low oxygen partial pressure just below the oxidation of iron at the elevated temperature of about 700 °C, that a significantly decreased oxidation behaviour for iron was observed at the surface of the Fe-Si alloys upon cooling, indicating a significantly enhanced oxygen uptake in that case [27].

As concerns the experiments of direct reduction of iron oxides, the high-temperature lab of the GO department is currently re-organized to meet the new requirements. For instance, while in the past the focus was on ultra-low mass changes at the surface and at grain boundaries, now the whole bulk of the sample shows a mass change, which for typical sample sizes is at the limit of our delicate high-sensitive equipment. On the other hand, the high mass sensitivity allows also to measure e.g. small cut single crystal iron oxides. First experiments with such single crystalline materials have been started. First results obtained on more classical pellets show that the relatively easy nucleation of magnetite and the subsequent wüstite as well as the fast solid-state diffusion (most likely inward diffusion of Fe^{2+}) through the iron oxide product layer are the main reasons for the fast reduction kinetics of the hematite to wüstite. The third step, viz. the reduction of wüstite into iron is – during the final 20 % reduction regime – nearly an order of magnitude slower. This effect can be most likely attributed to the sluggish mass transport (particularly of the outbound O solid-state diffusion) through the already formed iron product layers surrounding the wüstite. Near-atomic scale chemical probing reveals the presence of nano-sized Fe-containing transient-state oxides and the accumulation of certain gangue elements i.e. Ti and Na at the metal/oxide interface, both of which are assumed to be another cause for the slow reduction kinetics at the late stages of the wüstite reduction [28].

Atomistic Modelling (S. Wippermann)

In order to surmount materials-related challenges in the context of sustainability, optoelectronics, energy conversion and storage it is of critical importance to understand, predict and control the precise mechanistic details of interfacial chemical reactions and their dynamics at the femtosecond time-scale. The "Atomistic Modelling" group develops and applies accurate simulation techniques in close collaboration with experiments to explore light-matter interaction in nanostructures, electrified interfaces between nanostructures and liquid electrolytes, and their ultrafast dynamics.

Quantum dot solids with targeted properties are of particular interest in this context. Colloidal nano-scale building blocks, e.g. nanocrystals, can be used as "*artificial atoms*" and assembled into quantum dot solids. These nanomaterials located at the borderline between molecules and solids feature intriguing exciton-electron-phonon dynamics, where light can directly affect their fundamental physical properties. Moreover, nanocrystal assemblies provide opportunities to create quantum materials with targeted properties with possible applications, e.g., as next-generation light absorbers for photovoltaics, photoelectrochemistry and power electronics. Fig. 6 shows a prototypical example of a quantum dot solid, consisting of semiconducting core/shell nanocrystals. Either organic molecules or chalcogenidometallalate complexes can be used as "conductive glue", linking the individual building blocks.

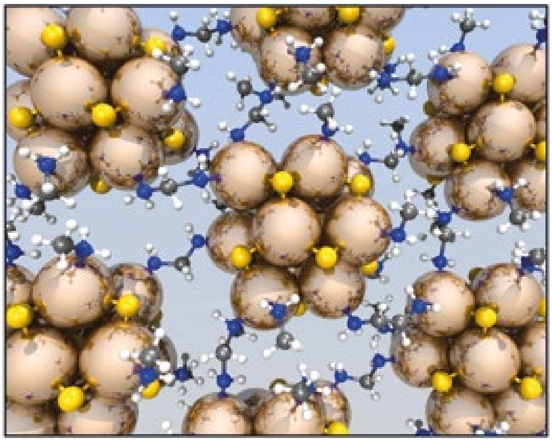


Fig. 6: *Schematic representation of a quantum dot solid, consisting of "magic-sized" nanocrystals assembled into a superlattice. Organic molecules or chalcogenidometallate complexes can be used as surface-selective linkers and "conductive glue", connecting the nanocrystals both mechanically and electrically.*

The Atomistic Modelling Group was awarded a NanoMatFutur-Grant from the Federal Ministry of Education and Research (BMBF) with a total value of 1.8 Mio. Euro 2014 to explore the nano-scale internal interfaces inherent to quantum dot solids and their impact on the emerging macro-scale electronic and optical properties. In addition to our existing collaboration with the Pritzker School for Molecular Engineering at the University of Chicago, two new collaborations were established within the current running period from 2018 – 2021 with the Institute of Basic Science Center for Nanoparticle Research and Center for Nanointegration at the University of Duisburg-Essen, respectively.

III-V semiconductor-based quantum dot solids, consisting of e.g. InP or InGaP nanocrystals, are among the most widely investigated materials systems due to their potentially superior optical properties and lower toxicity, compared to commercially available II-VI quantum dot solids. Whereas narrow emission line widths are readily achieved in II-VI quantum dot solids, III-V systems still suffer from inferior colour purity due to broad emissivity and poor photoluminescence quantum yields. We recently demonstrated [29] that nanocrystal stoichiometry dispersion is a major source of trap states and largely responsible for the observed emission broadening. Whereas II-VI quantum dot solids require only nanocrystal size control to achieve narrow emission, it is necessary to realize in addition nanocrystal stoichiometry control in the case of III-V quantum dot solids.

Anomalously strong temperature-induced shifts of the electronic bandgaps are another important source of emission broadening. In order to reveal the origin of these anomalous shifts, we used so-called magic-sized nanoclusters as atomically precise model systems for nanocrystals with zero dispersion in both size and stoichiometry. Tantalizingly, approaches that are commonly employed to accurately calculate temperature-dependent bandgaps in bulk materials have been known to predict qualitatively wrong results for nanocrystals since more than a decade ago. We conjectured that these shortcomings of traditional approaches are caused by their inability to take exciton-phonon coupling into account. We therefore developed a novel approach that allows us to accurately compute temperature-induced band gap shifts using constrained ab initio molecular dynamics (AIMD) simulations on the excited state potential energy surface and atomistic thermodynamics, where the band gap is computed as the Gibbs free energy of exciton formation. In contrast to existing methods, our approach explicitly takes the fully anharmonic exciton-phonon coupling into account.

We revealed [30] that the experimentally observed anomalous colour shifts with temperature are caused by excitonic bond-softening: in nanocrystals, the excitons are distributed over only a small number of atoms due to the strong quantum confinement. In consequence, the occupation of antibonding states due to the presence of the excitons significantly weakens the interatomic bonds, leading to a pronounced exciton-induced red shift of the phonon density of states (PDOS). This shift is almost two orders of magnitudes larger than the corresponding one observed in bulk materials. The red-shifted PDOS in turn is responsible for the anomalous bandgap shift due to its impact on the Gibbs free energy of exciton formation. Our results underline the importance of explicitly considering exciton-electron-phonon coupling in nanostructures and suggest how to design systems with improved optical properties.

Another fascinating example of ultrafast light-matter interactions is the precise optical control of structural transitions at the “quantum limit” in the regime of directed and deterministic nuclear motion. Chemical composition, pressure and temperature are commonly utilized to control and tune the properties of materials. Beyond these established concepts, here we explore how intense light fields and coherent control schemes inspired by concepts from femtochemistry can be used to guide the nuclear and electronic degrees of freedom through their complex multi-dimensional potential energy landscape into new states of matter.

To this purpose, the Atomistic Modelling Group established a new collaboration between the DFG FOR 1700 research group (see p. 93), the Institute of Basic Science Center for Low Dimensional Electronic Systems and the Max Planck Institute for Biophysical Systems. We use the atomic wire array known to form on the Si(111)-(4x1) In surface as an atomically precise model system in order to develop techniques for observing and controlling chemical bond-making and bond-breaking processes at the femtosecond time scale. Each atomic wire on this surface consists of two coupled monoatomic chains. At temperatures below T_C = 120 K, both chains undergo a period-doubling metal-insulator transition into a charge density wave (CDW) phase, driven by a triple-band Peierls instability. Thereby, formerly delocalized metallic states now become localized and form covalent bonds. This transition is of 1st order, hampered by an energy barrier. We demonstrated that the insulating and the metallic phases are able to coexist at temperatures close to T_C, giving rise to a phenomenon called electronic phase separation (EPS) and the formation of novel types of interfaces, such as metal-CDW junctions [31].

The transition itself is driven by multiple soft phonons with a strong coupling between electronic and lattice degrees of freedom. Due to the coupling between the two monoatomic chains, different superpositions of these phonons transform the chains into structurally distinct but energetically degenerate CDW states. Moreover, the coupling introduces a band inversion. As a consequence, the CDW phase supports fundamental excitations that take the form of interfaces between these degenerate states, and that feature topological properties with associated electronic chiral edge states inside the CDW band gap. In analogy to knots in a string, these topological states can be used to store and even process information at the molecular scale.

For all these reasons, atomic wires are promising targets in order to (i) explore the dynamics of chemical bond formation at the femtosecond time scale and (ii) develop, implement and test coherent optical control schemes using femtosecond laser pulse sequences. Based on our constrained AIMD approach, we design suitable pulse sequences and simulate the induced reaction dynamics. Depending on the targeted optical transition, a specific CDW state can be transformed e.g. into another structurally distinct CDW state or a supercooled metallic state. Fig. 7 shows a schematic outline of these concepts.

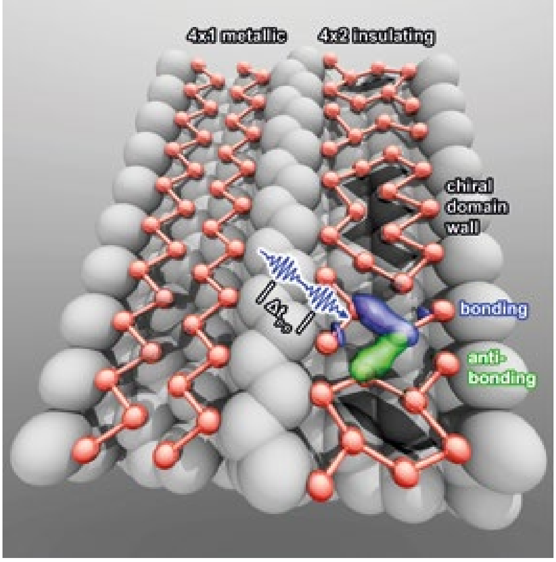


Fig. 7: *Schematic representation of structural motifs observed in the atomic wire array formed at the Si(111)-In surface. At room temperature, each indium wire consists of two coupled monoatomic indium zigzag chains. Below T_C = 120 K, the wires undergo a period doubling triple band Peierls transition into one of multiple possible charge density wave (CDW) states. The coupling between the monoatomic chains induces an electronic band inversion, leading to the formation of chiral topological edge states at domain boundaries between different CDW states. Exciting specific optical transitions by fs laserpulse sequences with carefully timed delays Δt_{P-P} allow us to form or dissolve specific chemical bonds.*

Extending these concepts, we recently started to explore the interaction of nanostructures with liquid electrolytes. Emerging energy conversion and storage technologies, such as artificial photosynthesis and power-to-X devices, critically depend on precisely engineering the chemical dynamics, reactivity and selectivity of interfaces between optically excited or electrified nanostructures and liquid electrolytes. Based on the Modern Theory of Polarization, we develop methods to incorporate electric fields in density-functional theory (DFT) and apply them to answer fundamental questions in the thermodynamics and transformation of electrochemical semiconductor-liquid interfaces.

In electrochemical experiments, the applied potential is held at a value that is constant on average at the macro-scale, but electronic and ionic charges fluctuate and transfer freely into and out of the region close to the interface, that is targeted by our simulations. In consequence, when studying elementary processes at electrified interfaces by DFT, we must treat the local surface charge not as a constant, but as a thermodynamic degree of freedom with temperature-dependent fluctuations. In collaboration with the CM department and building on our finite electric field techniques, we recently introduced a “thermopotentiostat”: a novel approach to control the electrode potential that can be used equally well to perform empirical or ab initio molecular dynamics simulations [32]. By design, (i) it satisfies the fluctuation-dissipation theorem exactly and is thus cleanly embedded into thermodynamic theory, (ii) requires only quantities that are either readily accessible in DFT codes or are known from the specific setup, and (iii) it is straightforward to include in any existing DFT code.

In order to demonstrate the performance of our thermopotentiostat approach, we considered a topic that had recently gained a lot of attention. Recent experiments performed in the A. Geim group (University of Manchester) showed that a water film confined to a few nm thickness changes its dielectric behaviour from the bulk dielectric constant of 80 down to 2. Thus, the presence of solid-water interfaces appears to modify the dielectric response of water from a highly polarizable medium, which is considered to be the origin of the unique solvation behaviour of water, down to a response that is close to the vacuum dielectric constant. Understanding and being able to qualitatively describe this mechanism is crucial, since interfacial water is omnipresent and electrochemical reactions in particular occur within the interfacial water region.

Because of the relevance of this question in fields as diverse as electrochemistry, corrosion and electrocatalysis, multiple computational studies addressed dielectric properties of nanoconfined water. These studies generally rely on Kirkwood-Fröhlich theory, using the variance of the total dipole moment fluctuations per volume, or on the theory of polarization fluctuations. However, the exact location of the boundary between the electrode and the dielectric is ill-defined. Past studies therefore reported only dipole fluctuations perpendicular to the electrode surface, but not the dielectric constant itself.

Our thermopotentiostat approach allows us to address this question directly, since our computational setup shown in Fig. 8a exactly reproduces the experimental situation. Here, liquid water is confined between two electrodes where the electrode charge is actively controlled by our thermopotentiostat. Fig. 8b shows the calculated static dielectric constant as a function of the water layer thickness, compared to the experimental data from the Geim group. Consistent with the measurements, our results display a pronounced decrease of the dielectric constant compared to the static dielectric constant of liquid bulk water, that persists for electrode separations exceeding 100 nm.

In order to understand the origin of the decreasing dielectric constant with decreasing electrode separation, we computed the local inverse static dielectric constant as a function of the normal distance to the electrode surface, cf. Fig. 8c. At the position of the electrode surface, the inverse dielectric constant drops sharply and intersects the water bulk value at ~ 3 Angstrom above the surface. With further increasing distance, it assumes negative values for interfacial water and then approaches the bulk water value in an oscillatory fashion. Beyond a normal distance of ~ 9 Angstrom, the dielectric constant of bulk liquid water is recovered. Our results demonstrate that the region with modified dielectric properties is closely confined to the region of interfacial water. Introducing a continuum surrogate model informed by the dielectric profile shown in Fig. 8c, we demonstrated that the local dielectric properties of water close to the interface are indeed responsible for the observed reduction of nanoconfined water’s

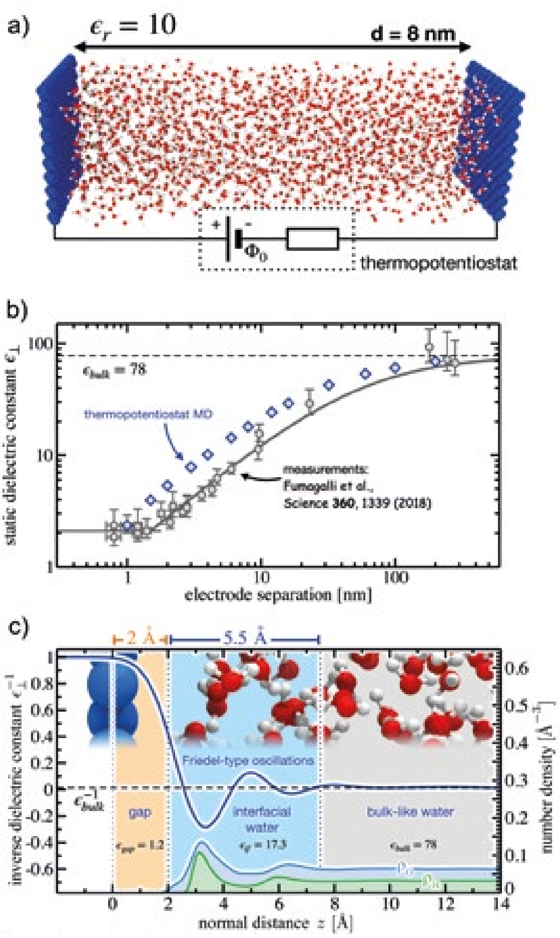


Fig. 8: ***a)*** *Computational setup, consisting of liquid water confined between two electrodes. The electrode charge is actively controlled by our thermopotentiostat.* ***b)*** *Static dielectric constant for TIP3P water as a function of electrode separation, calculated using thermopotentiostat molecular dynamics (MD). Quantitative differences with respect to the measurements encode chemical information on the specific surfaces used in the experiments, as our model is based on generic hydrophobic electrodes. Experimental data reproduced with permission from Science 360 (2018) 1339.* ***c)*** *Local inverse dielectric profile and O/H number density profiles. Reproduced with permission from the American Physical Society [4].*

dielectric constant compared to the static dielectric constant of bulk water [32].

This intriguing behaviour of the polarization density within interfacial water resembles the physics of Friedel oscillations at metallic surfaces, albeit with a much stronger amplitude. The wavelength of the oscillations corresponds to the size of the water molecules. A negative local dielectric constant implies a net attractive force between like charges. On-going research builds on these findings to explore the formation and structure of electrochemical double layers and charge transfer reactions at electrified solid-water interfaces.

We note that the calculation of dielectric profiles from polarization fluctuations requires hundreds of nanoseconds of statistical sampling, in practice enforcing the use of classical molecular dynamics. In contrast, the use of stochastic canonical sampling in our thermopotentiostat technique in conjunction with finite electric field methods allows us to rely purely on thermodynamic averages rather than variances. Thus, the required computational time to converge the dielectric profiles is reduced by more than two orders of magnitude, placing these types of calculations now well within reach of *ab initio* molecular dynamics simulations.

These research activities focus on solving outstanding fundamental open problems in the context of light-matter interaction in nanostructures, low dimensional electronic systems and electrified interfaces.

Our activities benefit from mutual insights obtained in different areas of interface chemistry and surface science research conducted at the MPIE. In order to foster also exchange at an international level, in February 2019 with the FOR 1700 research group (see p. 93). we conducted a three-day workshop at Ringberg Castle on “Surface Science: The Past, Present and Future”, in order to bring together leading scientists from the areas of low dimensional electronic systems, surface spectroscopy and strongly correlated systems. For April 2020, together with the CM department we planned a three-day workshop at Ringberg Castle on “Electrified solid/water interfaces – theory meets experiment”, inviting leading scientists from the fields of electrochemistry, solvation and spectroscopy. Due to the CoViD-19 situation, this workshop has been rescheduled to March 2022.

Interface Spectroscopy (A. Erbe)

This group was active as a guest group until end of 2019. Strong activities of that group were in the field of optical methods development. In collaboration with the former research group “Interaction Forces and Functional Materials” of M. Valtiner (now professor at TU Wien, Austria) a novel method for the analysis of data acquired in a typical surface force apparatus experiment has been developed. This method adapted known matrix methods for analysing reflectivity spectra with the specific requirements of the optics of surface force apparatus measurements. This method helps to overcome limitations in the approximations of classical methods [33].

In a collaboration between the MPIE, the University of Pisa, the Italian National Research Council and the Indian Institute of Technology Dhanbad, a novel microwave-based polymerisation technique was used to make poly(styrene) microspheres with few µm diameter that contained a lasing dye. The resulting particles have been shown to be amongst the world’s smallest polymer-based whispering gallery mode supporting micro-resonators, and are suitable for sensing the environment at the particle / solution interface via changes in the interfacial refractive index [34].

In a collaboration with the Norwegian University of Science and Technology, infrared imaging was used to image subsurface modifications of silicon, which were prepared by the partner with a non-linear optical laser writing technique [35].

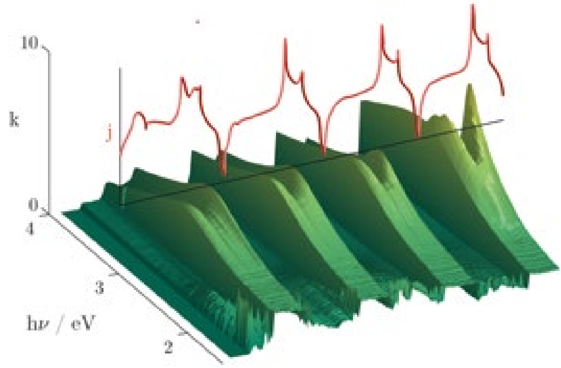


Fig. 9: *Results of in situ electrochemical spectroscopic ellipsometry. The positive/negative peaks in the current density (j) indicate formation/reduction of thin oxide layers. The measured k-spectra illustrated as 3D plot indicate reversible changes in the electronic structure at the onset of the oxygen evolution reaction.*

Another topic was the Application of spectroscopic techniques for investigation of electrochemical and corrosion phenomena. Traditionally, the “Interface Spectroscopy” group has used optical techniques for materials characterisation. In the search for novel abundant catalytic materials for electrochemical water splitting the oxide layer growth on manganese during electrochemical oxidation and oxygen evolution reaction (OER) in basic electrolyte was thoroughly studied, in cooperation with M. Rabe [36]. Using Raman and ellipsometric in situ spectroscopy stable oxide films containing mostly α-MnO2 were observed at the onset of OER (Fig. 9). High Mn dissolution rates were measured during OER, limiting the applicability as catalyst. However, still a stationary stable, highly disordered phase was found, containing MnO_6 tetrahedra. In a PhD thesis, which was finalised in the reporting period, zinc corrosion was investigated in detail by spectroscopic methods with dif-

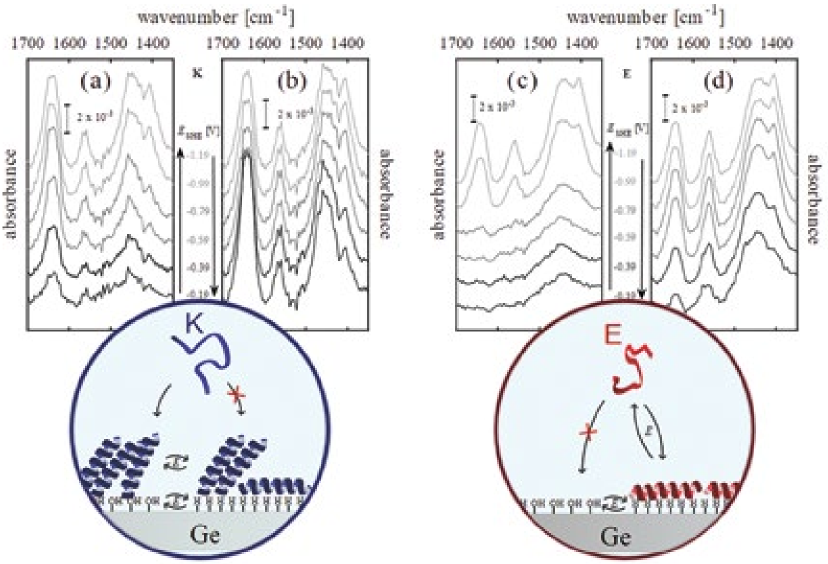


Fig. 10: *Results of in situ ATR-IR spectroscopy to study electro-responsive smart Ge interfaces. The detailed analysis of potential dependent amide I' peaks (at ca. 1650 cm^{-1}) and the Ge-D stretching band (shoulder at 1405 cm^{-1}) in the spectra (a-d) revealed mechanistic details of reversible adsorption and orientation changes of the helical model peptides.*

ferent collaborators. Besides an imaging of the spatial distribution of the corrosion products, the detection of a hydrogen saturated oxygen vacancy as an important defect in the initial corrosion products of zinc in sea water stands out [37].

Finally, also the work within this group on Surface chemistry of reactive materials should be mentioned. As a side result of a project on zinc pre-treatment with biopolymer based coatings, a switch in the surface appearance between black and white when changing the electrode potential of zinc coated with a gelatine-based coating was achieved [38].

In a study of the surface modification of copper with organic alkyl thiols, solvent specific adsorbate structures were found. In particular, the C-S bond in the thiol molecule was found to be cleaved through the strong Cu-S interaction [39]. As part of a systematic study of the interaction of model organic coatings with metal substrates, the cathodic delamination rates of differently surface bound poly(styrene) coatings have been compared [40]. Healing of coating defects upon damage has been an important focus of the work in the department. In one project, the inclusion into a pre-treatment of a cyclic oligosaccharide incorporating a film-forming corrosion inhibitor has been shown to lead to healing of defects in an organic model coating on zinc [12].

Spectroscopy at Electrochemical Interfaces (M. Rabe)

The early career research group "Spectroscopy at Electrochemical Interfaces" was newly set up in 2020 and is co-funded by the MPIE and the cluster of excellence 'Ruhr explores solvation' RESOLV (see p. 86). It partly stands in the tradition of the "Optical Spectroscopy Group" of A. Erbe, and now develops a new independent research profile. In the group optical interface spectroscopy methods are employed for exploring interface characteristics and dynamic processes. Ongoing and future research activities specifically focus on interface solvation, a fundamental phenomenon occurring whenever a liquid phase -especially water- terminates at a solid interface. This specific structural arrangement of solvent molecules on interfaces plays vital roles for instance in electrochemical applications, biological reactions or atmospheric processes, which require a fundamental understanding. Furthermore, electrochemical *in situ* spectroscopy is applied for the study of OER catalysts employed in alkaline water electrolysis for the production of green hydrogen (for more details on future and ongoing projects see p. 45).

In several cooperation projects plain or layer covered metallic substrates or alloys were studied employing the expertise in spectroscopic ellipsometry (SE). In a collaboration with the Faculty of Chemistry of Silesian University of Technology in Gliwice, Poland, conversion coatings formed by anodization in alcohols on top of galvanic Zn-Ni alloy coatings on steel were studied [41]. The coatings were composed of mixed oxide-alkoxides and featured high corrosion resistance and optical colouration. The detailed analysis of the SE data revealed that the visible colour varies with the layer thickness of the coating, which was a mixed oxide-alkoxide layer and can be controlled by the anodization process.

In an internal cooperation with the "Corrosion" group the formation of water layers on various noble metals in humidified atmospheres up to 98 % rh are examined. For instance, the layers formed under these circumstances on Pd are ultrathin < 1 nm [1], but electrochemical reactions underneath such layers play important roles in technically relevant processes such as electrolysis, fuel cells and atmospheric corrosion. Currently, similar studies on Ir and Au are ongoing.

In a project collaboration with the VDEh-Betriebsforschungsinstitut GmbH (BFI), the optical properties of steel samples of varying composition are studied by SE. Aim of the BFI-led project is to develop a system for the in-line measurement of the steel composition directly in the liquid melt by reflection spectroscopy. Interestingly, variation of the concentrations of specific elements such as Ni and Cr have been found to have a significant influence on the reflectivity of the steel samples at normal incidence, which supposedly can be exploited for the planned measurement principle. The technical development and pilot implementation at BFI is ongoing and further supported by our group.

In order to understand and design electro responsive ‘smart’ interfaces, research has been conducted exploring the interactions of α-helical peptides with interfaces. Following up on earlier work, performed in the GO department that revealed details of the electrochemically triggered reduction of germanium surface termination, this reversible termination change was employed for a hydrophobic/hydrophilic switchable, ‘smart’ interface. By employing *in situ* attenuated total reflection infrared (ATR-IR) spectroscopy it was shown that the hydrophobicity switch can reversibly trigger ad-/desorption as well as orientation change of amphipathic model peptides (Fig. 10). The observed process (ad-/desorption or orientation change) depends on the adsorbates charge distribution, which indicates that a sensitive balance between hydrophobic and electrostatic interactions governs the surface attachment.

Chemometric methods are developed and implemented in the group to improve the analysis of complex or large multivariate spectroscopic data. A hard modelling multivariate curve resolution (MCR) approach was implemented to resolve multivariate spectral data with several components lying closely together in terms of their peak positions (as function of photon energy or wavenumber), but with individual dependencies on a specific control variable (such as temperature, pH, potential, etc.). The approach is based on empirical or physical transition models chosen by the analyst that describe the dependency of the spectral intensity on the control variable. Within several research projects in the GO department, this approach has been employed and advanced. For instance, for the analysis of *in situ* ATR-IR spectra of germanium desolvation and photoluminescence spectra of corrosion products on zinc [37]. Most recently, the implementation was extended to allow a convenient application to complex data sets with virtually unlimited spectral components and an individual choice of transition models. This development significantly eases the application of the method for different spectroscopic methods in diverse fields. It is available as open source library [42]. In a collaboration with Leiden University a method was developed and implemented for the analysis of multivariate titration curves of metal binding of multicore peptide-metal complexes. By this method circular dichroism spectroscopy data was analysed to examine the thermodynamics of folding of the trinuclear Ni(II) and Cu(II) complexes of the designed α-helical peptide trimer HisAD [43]. Unexpectedly, it was found that the titration data can only be explained by a 3-state model and it was shown that this is caused by a mononuclear intermediate in the folding pathway.

Dynamic Electrocatalytic Interfaces (O. Kasian)

This is the former group of K. Mayrhofer who left the MPIE already in 2015, but which was still operating until now, first still headed by K. Mayrhofer and since 2019 by O. Kasian. O. Kasian holds the position of head of a

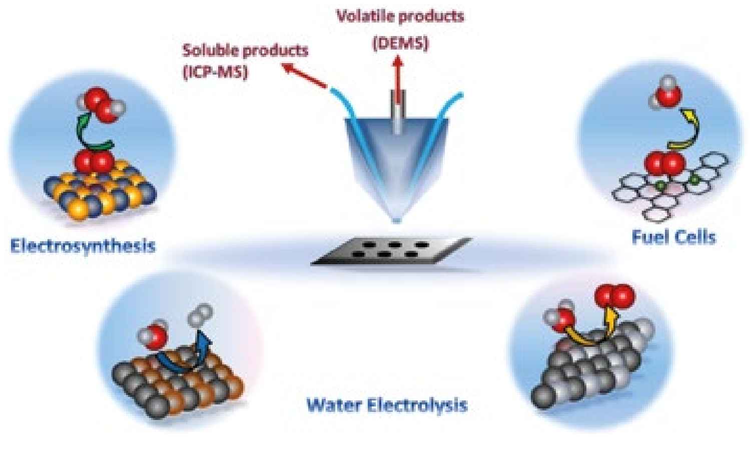


Fig. 11: *Schematic overview of the applications of a flow cell coupled to analytics for simultaneous detection of soluble and volatile reaction products and intermediates in energy conversion related applications such as fuel cells and electrolysis. On-line ICP-MS (inductively coupled plasma mass-spectrometry) allows precise quantification of dissolution in electrochemistry, while DEMS (differential electrochemical mass spectrometry) enables qualitative analysis of gaseous species formed during the reaction.*

Young Investigator Group at the Helmholtz Institute Berlin in collaboration with the Helmholtz Institute Erlangen-Nürnberg for Renewable Energy. At the MPIE she was offered to head the guest group on “Electrocatalysis”. The research is focused on the dynamic transformations of the catalytic surfaces under the reaction conditions. The group develops nanostructured model thin film surfaces and explores their structural and compositional changes induced by the catalytic processes and the effect of such transformations on the mechanism and kinetics of the reactions in electrocatalysis. This is achieved by combination of various *ex situ* and *in situ* methods.

In fact, until 2020 a part of the research was still focussed on K. Mayrhofer´s activities, such as on oxygen reduction [44, 45], hydrogen peroxide synthesis [46], CO_2 reduction [47], stability of catalysts [45, 48, 49]. hydrogen evolution reaction [20] and oxygen evolution reaction [50].

The activities of O. Kasian at the MPIE were mainly related to water splitting.

Understanding the fundamentals of electrocatalytic reactions is of importance for the development of efficient and durable energy conversion and storage devices. In such devices the target reactions are often accompanied by unwanted degradation or deactivation of the catalysts. These reactions are often ongoing via formation of common intermediates, which makes mechanistic studies very complex and requires employment of highly sensitive analytics. In the “Dynamic Electrocatalytic Interfaces ” group we have developed an approach to distinguish between intermediates and products of various reactions combining isotope labelling with online electrochemical and inductively coupled plasma mass spectrometry [51 and Fig.11].

Recently, employing this technique the group aided in understanding of the water splitting mechanism on iridium-based oxides [51-54], a pivotal and timely topic in the ‘green’ hydrogen production technology. Iridium oxide anodes are used to catalyse formation of oxygen from water, because of their superior corrosion resistance and relatively high catalytic activity. Nevertheless, even iridium undergoes dissolution in the harsh conditions of the oxygen evolution. This is especially crucial for catalysts with high reactivity, such as hydrous oxides. Our approach allowed us to provide a quantitative assessment of the degree of lattice oxygen participation in the oxygen evolution reaction and the associated degradation of the Ir-oxides. The data obtained on ^{18}O-labeled catalysts reveal that amorphous hydrous oxides are prone to degradation via so-called ‘lattice oxygen evolution mechanism’, in which oxygen molecule can be formed directly from the oxide lattice atoms without water discharge. This oxygen evolution mechanism results in faster degradation and dissolution of Ir. In compact oxide structures, e.g. rutile IrO_2 such mechanism is less probable.

Overall our results provide a contribution to the fundamental understanding of the exceptional stability of Ir-oxides towards the oxygen evolution reaction. The proposed approach to a quantitative assessment of the degree of lattice oxygen participation in the oxygen evolution reaction can be further applied to other state-of-the-art catalyst systems used in fuel cells or electrolysers.

References

1. Zhong, X.K.; Schulz, M.; Wu, C.H.; Rabe, M.; Erbe, A.; Rohwerder, M.: Chemelectrochem. 8 (2021) 712.
2. Azzam, W.; Al-Rashdi, A.; Subaihi, A.; Rohwerder, M.; Zharnikov, M.; Bashir, A.: Phys. Chem. Chem. Phys. 22 (2020) 13580.
3. Azzam, W.; Sauter, E.; Alrashdi, A.A.; Al-Refaie, N.; Rohwerder, M.; Bashir, A.; Zharnikov, M.: J. Phys. Chem. C 123 (2019) 4308.
4. Fu, Z.H.; Ladnorg, T.; Gliemann, H.; Welle, A.; Bashir, A.; Rohwerder, M.; Zhang, Q.; Schüpbach, B.; Terfort, A.; Wöll, C.H.: Beilstein J. Nanotechnol. 10 (2019) 2449.
5. Shkirskiy, V.; Uebel, M.; Maltseva, A.; Lefèvre, G.; Volovitch, P.; Rohwerder, M.: npj Mat. Degrad. 3 (2019) 2.
6. Prabhakar, J.M.; Varanasi, R.S.; da Silva, C.C.; Saba; de Vooys, A.; Erbe, A.; Rohwerder, M.: Corros. Sci. 187 (2021) 109525.
7. Merz, A.; Uebel, M.; Rohwerder, M.: J. Electrochem. Soc. 166 (2019) C304.
8. Merz, A.; Rohwerder, M.: J. Electrochem. Soc. 166 (2019) C314.
9. Exbrayat, L.; Salaluk, S.; Uebel, M.; Jenjob, R.; Rameau, B.; Koynov, K., Landfester, K.; Rohwerder, M.; Crespy, D.: ACS Appl. Nano Mater. 2 (2019) 812.
10. Salaluk, S.; Jiang, S.; Viyanit, E.; Rohwerder, M.; Landfester, K.; Crespy, D.: ACS Appl. Mater. Interfaces 13 (2021) 53046.
11. Yin, Y.; Schulz, M.; Rohwerder, M.: Corros. Sci. 190 (2021) 109661.
12. Altin, A.; Vimalanandan, A.; Sarfraz, A.; Rohwerder, M.; Erbe, A.: Langmuir 35 (2019) 70.
13. Peng, J.; Moszner, F.; Rechmann, J.; Vogel, D.; Palm, M.; Rohwerder, M.: Corros. Sci. 149 (2019) 123.
14. Springer, H.; Baron, C.; Tanure, L.; Rohwerder, M.: Mater. Today Commun. 29 (2021) 102947.
15. Fenker, M.; Balzer, M.; Kappl, H.; Heyn, A.; Rohwerder, M.; Fink, N.; Mingers, A.: Thin Solid Films 688 (2019) 137430.
16. Gohl, D.; Ruess, H.; Schlicht, S.; Vogel, A.; Rohwerder, M.; Mayrhofer, K.J.J.; Bachmann, J.; Roman-Leshkov, Y.; Schneider, J.M.; Ledendecker, M.: Chemelectrochem 7 (2020) 2404.
17. Aguirre, J.; Walczak, M.; Rohwerder, M.: Wear 438 (2019).
18. Gerlitzky, C.; Kuhn, C.; Groche, P.; Tran, T.H.; Zhang, S.; Peter, N.J.; Rohwerder, M.: Materialwiss. Werkst. 50 (2019) 913.
19. Tran, T.H.; Gerlitzky, C.; Rohwerder, M.; Groche, P.: Which Properties Must a Surface have to be Suitable for Cold Pressure Welding?, in: Galdos, L.; Arrazola, P.; DeArgandona, E.S., et al. (ed.), Proceedings of the 22nd International Esaform Conference on Material Forming (2019) 050019.
20. Zeradjanin, A.R.; Polymeros, G.; Toparli, C.; Ledendecker, M.; Hodnik, N.; Erbe, A.; Rohwerder, M.; La Mantia, F.: Phys. Chem. Chem. Phys. 22 (2020) 8768.
21. Zeradjanin, A.R.; Spanos, I.; Masa, J.; Rohwerder, M.; Schlogl, R.: J. Solid State Electrochem. 25 (2020) 33.
22. Sun, B.H.; Krieger, W.; Rohwerder, M.; Ponge, D.; Raabe, D.: Acta Mater. 183 (2020) 313.
23. Wu, C.H.; Krieger, W.; Rohwerder, M.: Sci. Technol. Adv. Mat. 20 (2019) 1073.

24. Mischke, J.; Pennings, J.; Weisenseel, E.; Kerger, P.; Rohwerder, M.; Mertin, W.; Bacher, G.: 2D Mater.7 (2020).
25. Zhang, X.; da Silva, C.C.; Zhang, S.; Prabhakar, M.; Lu, W.; Vogel, A.; Rohwerder, M.: Corros. Sci. 186 (2021) 109466.
26. Zhang, X.; Corrêa da Silva, C.; Liu, C.; Prabhakar, M.; Rohwerder, M.: Corros. Sci. 174 (2020) 108859.
27. Silva, C.d.: Investigation of the kinetics of selective oxidation of iron model alloys during simulated annealing, Doctoral Thesis, Ruhr Universität Bochum (2020).
28. Kim, S.-H.; Zhang, X.; Ma, Y.; Souza Filho, I.R.; Schweinar, K.; Angenendt, K., Vogel, D.; Stephenson, L.; El-Zoka, A.; Mianroodi, J.R.; Rohwerder, M.; Gault, B.; Raabe, D.: Acta Mater. 212 (2021) 116933.
29. Rusishvili, M.; Wippermann, S.; Talapin, D.V.; Galli, G.: Chem. Mater. 32 (2020) 9798.
30. Muckel, F.; Lorenz, S.; Yang, J.; Nugraha, T.A.; Scalise, E.; Hyeon, T.; Wippermann, S.; Bacher, G.: Nat. Commun. 11 (2020) 4127.
31. Song, S.K.; Samad, A.; Wippermann, S.; Yeom, H.W.: Nano Lett. 19 (2019) 5769.
32. Deissenbeck, F.; Freysoldt, C.; Todorova, M.; Neugebauer, J.; Wippermann, S.: Phys. Rev. Lett. 126 (2021).
33. Schwenzfeier, K.A.; Erbe, A.; Bilotto, P.; Lengauer, M.; Merola, C.; Cheng, H.W.; Mears, L.L.E.; Valtiner, M.: Rev. Sci. Instrum. 90 (2019) 043908.
34. Mondragón-Ochoa, J.S.; González-Rivera, J.; Toparli, C.; Khanum, R.; Moirangthem, R.S.; Duce, C., Ferrari, C.; Barillaro, G.; Erbe, A. : J. Phys. D: Appl. Phys. 55 (2021) 055101.
35. Richter, R.A.; Tolstik, N.; Rigaud, S.; Dalla Valle, P.; Erbe, A.; Ebbinghaus, P., Astrauskas, I.; Kalashnikov, V.; Sorokin, E.; Sorokina, I.T.: J. Opt. Soc. Am. B 37 (2020) 2543.
36. Rabe, M.; Toparli, C.; Chen, Y.H.; Kasian, O.; Mayrhofer, K.J.J.; Erbe, A.: Phys. Chem. Chem.Phys. 21 (2019) 10457.
37. Tecklenburg, S.: Defect formation and evolution in zinc oxide: from semiconductors to corrosion, Doctoral Thesis, Ruhr Universität Bochum (2019).
38. Ksiazkiewicz, A.N.; Fernandez-Solis, C.; Erbe, A.: J. Phys. Materials 3 (2020) 025009.
39. Rechmann, J.; Krzywiecki, M.; Erbe, A.: Langmuir 35 (2019) 6888.
40. Iqbal, D.; Rechmann, J.; Bashir, A.; Sarfraz, A.; Altin, A.; Erbe, A.: Mater. Corros . 70 (2019) 481.
41. Maciej, A.; Wadas, A.; Sowa, M.; Socha, R.; Dercz, G.; Rabe, M.; Simka, W.: Corros. Sci. 158 (2019) 108107.
42. Rabe, M.: J. Open Res. Softw. 8 (2020) 13.
43. Boyle, A.L.; Rabe, M.; Crone, N.S.A.; Rhys, G.G.; Soler, N.; Voskamp, P.; Pannu, N.S.; Kros, A.: Chem. Sci. 10 (2019) 7456.
44. Fortunato, G.V.; Pizzutilo, E.; Cardoso, E.S.F.; Lanza, M.R.V.; Katsounaros, I.; Freakley, S.J., Mayrhofer, K.J.J.; Maia, G.; Ledendecker, M.: J. Catal. 389 (2020) 400.
45. Gohl, D.; Ruess, H.; Schlicht, S.; Vogel, A.; Rohwerder, M.; Mayrhofer, K.J.J., et al.: Chemelectrochem 7 (2020) 2404.
46. Ledendecker, M.; Pizzutilo, E.; Malta, G.; Fortunato, G.V.; Mayrhofer, K.J.J.; Hutchings, G.J.; Freakley, S.J.: ACS Catal. 10 (2020) 5928.
47. Osowiecki, W.T.; Nussbaum, J.J.; Kamat, G.A.; Katsoukis, G.; Ledendecker, M.; Frei, H.; Bell, A.T.; Alivisatos, A.P.: ACS Appl. Energy Mater. 2 (2019) 7744.
48. Gohl, D.; Garg, A.; Paciok, P.; Mayrhofer, K.J.J.; Heggen, M.; Shao-Horn, Y., Dunin-Borkowski, R.E.; Roman-Leshkov, Y.; Ledendecker, M.: Nat. Mater. 19 (2020) 287.
49. Gohl, D.; Ruess, H.; Pander, M.; Zeradjanin, A.R.; Mayrhofer, K.J.J.; Schneider, J.M.; Erbe, A.; Ledendecker, M.: J. Electrochem. Soc. 167 (2020).
50. Ledendecker, M.; Geiger, S.; Hengge, K.; Lim, J.; Cherevko, S.; Mingers, A.M., Göhl, D.; Fortunato, G.V.; Jalalpoor, D.; Schüth, F.; Scheu, C.; Mayrhofer K.J.J.: Nano Res. 12 (2019) 2275.
51. Kasian, O.; Geiger, S.; Li, T.; Grote, J.P.; Schweinar, K.; Zhang, S.Y., Scheu, C.; Raabe, D.; Cherevko, S.; Gault, B.; Mayrhofer, K.J.J.: Energy Environ. Sci.12 (2019) 3548.
52. Kasian, O.; Li, T.; Mingers, A.M.; Schweinar, K.; Savan, A.; Ludwig, A.; Mayrhofer, K.: J. Phys. Energy 3 (2021).
53. Schweinar, K.; Gault, B.; Mouton, I.; Kasian, O.: J. Phys. Chem. Lett. 11 (2020) 5008.
54. Kasian, O.; Geiger, S.; Mayrhofer, K.J.J.; Cherevko, S.: Chem. Rec. 19 (2019) 2130.

Department of Microstructure Physics and Alloy Design (MA)

D. Raabe

Introduction

Scientific mission

We study microstructures and their influence on the properties of materials, mostly metals. Microstructure comprises the structure, size, dimension, patterning, and chemistry of all lattice defects including vacancies, dislocations, and interfaces. We study individual defects such as single vacancies (with Field Ion Microscopy and atomistic modelling) [1–3] and also large statistical defect ensembles such as light years of entangled dislocation lines (for instance by X-ray diffraction and crystal plasticity modelling) [4–7].

Microstructure affects all materials, from pure single crystals [8, 9] to complex engineering alloys [10–13]. It can change, by orders of magnitude, a material's mechanical behaviour (e.g. strength, ductility), electrochemical response (e.g. corrosion, charging behaviour), and functional properties (e.g. magnetic hysteresis, electrical conductivity).

For tailoring microstructures and chemistry, we work on alloy design [14–18], metallurgical processing [19–21], combinatorial synthesis [21–25], sustainable production [26–29], and additive manufacturing [30–36].

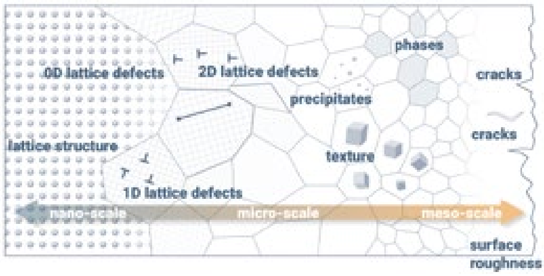


Fig. 1: *Microstructure cosmos, consisting of a range of lattice defects and their chemical decoration features.*

Our main analysis tools are computational materials science [37–39], machine learning [3, 13, 40–42] as well as multiscale and multi-probe characterization [18, 43–49].

With this expertise we have developed a knowledge-based as opposed to the traditional try-and-error-approach to the development of new materials, microstructures, processes, and property combinations, often by reconciling sometimes antagonistic features such as mechanical strength, ductility and soft magnetic behaviours [20, 50–54].

Impact on society

Since the dawn of mankind, materials have been the backbone of human society. Today, they are indispensable in the fields of energy, industry, transport, health, construction, safety, and manufacturing. With >2 billion tons produced every year, metals stand for massive economic growth, job safety, and wealth increase. Due to the sheer quantities produced and used, they also play a central role in sustainability.

Currently, we enter from the age of linear industry into a circular and digitalized economy. This offers huge opportunities to revolutionize production, transport, and energy supply. These changes affect the daily lives of billions of people. Advanced metallic alloys, their production, use, and recycling are key to this transition, as they can help enabling a carbon-free, digitalized and electrified industrial and urban future.

Metals play a twofold role in that context. On the one hand they enable many products and processes through which energy can be saved and greenhouse gas emissions can be reduced. Examples are thermoelectrics for waste heat harvesting [55–59], high-strength alloys for reducing the weight of vehicles we discuss here cases where metastable phases are not coincidentally inherited from processing, but rather are engineered. Specifically, we aim at compositional (partitioning [20, 60–63], or magnetic materials [54, 64–66] for electrical engines. On the other hand, they are the highest single source of greenhouse gas emissions, particularly due to synthesis, which often involves carbon as a reductant, making metallurgy the biggest single cause for global warming [27–29].

Therefore, we devote our efforts to understand, invent, and enable advanced materials and processes for a sustainable and safe future [26–29].

Fields of Special Methodological Expertise

Computational materials science

Our expertise in this field lies in the theory of micromechanics and its non-linear interplay with chemistry and phase transformations [5, 55–58]. Recently, we have also started to include the interaction of micromechanics and redox reactions in our simulations, such as needed to describe material decay, corrosion, and reduction processes in energy conversion applications, harsh environmental conditions and metallurgical sustainability.

Fig. 2: *Microstructure simulation considering mechanics, chemistry, phase transformations, and redox reactions.*

We have cast our research experience and simulation codes accumulated over more than 25 years into the software package DAMASK [5, 37, 59–62], a free material simulation kit for modelling multi-physics crystal plasticity, thermal, phase transformation, and damage phenomena from the single crystal up to the component scale (Fig. 2, see also p. 73). It is employed by hundreds of academic users and companies worldwide (https://damask.mpie.de). We use it for both, predicting microstructure evolution and the resulting properties on the one hand and for boundary condition treatment of experiments on the other hand. This approach enables us to render microstructure research quantitatively.

Multiscale and multi-probe microstructure characterization

Our expertise in this domain lies in applying fully cor-relative atomic-scale and mesoscale probing of struc-ture, defects and chemistry to the exactly same mate-rial position, to jointly probe structural, chemical, and property features e.g. of chemical decoration and transformation phenomena at internal interfaces, environmental degradation, dislocations, or even single vacancies[18, 43–49]. We use combinations of atom probe tomography (APT) [63, 64], electron channelling contrast imaging under controlled diffraction condi-tions (ECCI) [65–68], 3D electron imaging and backscatter diffraction (EBSD) [69–72], cross-correlation EBSD [73, 74], and field ion microscopy (FIM) [1–3], often coupled with theory and machine learning, for enhanced quantification of the data. We apply such correlative multi-probe methods to a range of materials, from well-defined single crystals up to complex engineering metallic materials. This approach enabled discoveries such as low-dimensional trans-formations of chemically decorated dislocation core regions [75, 76], interfacial spinodal-like chemical decomposition effects at interfaces [12, 77, 78], or partitioning phenomena [10, 79–82] among several types of adjacent phases and lattice defects.

Increasing focus in characterisation lies in considering environmental degradation of materials. This is a particularly challenging task when it comes to the joint characterization of microstructures, damage features, and the reactants and reactions that are causing the degradation, involving for instance hydrogen (to study hydrogen embrittlement), oxygen (to study corrosion), and light reactive elements such as lithium (to study batteries) [13, 26, 45, 46, 83–85]. Especially hydrogen is extremely hard to image and once being mapped one must make sure that it is the hydrogen that was charged in an experiment by purpose and not the hydrogen that had intruded due to contaminated experimental conditions. We have therefore established a cryogenic and ultra-high vacuum workflow that combines a reaction and charging laboratory hub with plasma focused ion beam, electron optical, and atom probe tomography characterization instruments (see p. 47 & 50).

Alloy and process design

In this fields we apply our approaches to make materials that have property profiles with often conflicting features. Examples are alloys with high mechanical strength on the one hand and good ductility, fracture toughness, thermoelectric performance, hydrogen embrittlement resistance, or functional properties on the other hand [14–18].

As one example to solve the fundamental conflict between strength and ductility we introduced a gener-alized metastability alloy design principle [14, 79, 86]. This means that the thermodynamic stability of the dominant matrix phase or of a chemically decorated confined region (such as a single lattice defect or interface region) of an alloy is compositionally adjusted in such a way that athermal transformation mecha-nisms (such as twinning and martensite formation which both profoundly increase the material’s strength) are activated in a specific window of the stress-strain regime where they are needed to coun-teract localization and micro-damage inside the alloy. This metastability alloy design concept has led to a number of discoveries of new materials with excep-tional load-bearing capacity paired with high damage tolerance [35, 54, 95, 97–100].

Another example is in magnetic alloys [12, 48, 87–89]: regarding the combination of strength and good func-tional properties we develop high strength soft mag-netic materials, for instance by utilising spinodal de-composition or alloys with high volume fraction of precipitates. Another direction lies in developing alloys and coatings with specific compositions and micro-structures that have a high resistance to hydrogen embrittlement and corrosion [26, 90–92].

Processes, mechanisms, and materials for sustainability and a circular economy

The microstructure-centred approach of the department enables us to address our latest research quest, namely, to identify pathways towards enhanced sustainability of metallic materials, in areas which include reduced-CO_2 primary production, recycling of metals, scrap-compatible alloy design, contaminant- and hydrogen tolerance of alloys, hydrogen-plasma based reduction, electrolysis for the reduction of oxides, and H-based direct reduction of iron ores [26–29].

For this purpose, we have designed and modified a number of laboratory-scale reactors in which corresponding experiments can be conducted, under well-controlled reactive boundary conditions and temperature control as well as permanent in-operando monitoring through mass spectrometry. These experiments are conducted in close cooperation with the group of M. Rohwerder at the GO department (see p. 125).

Some topics are currently pursued with high activity, namely, H-based direct reduction of iron oxides, H-plasma-based reduction (see p. Ma group), and the design of alloys that can tolerate highest possible scrap and thus impurity fractions.

These projects show two interesting trends, a more specific and a more general one: Firstly, there are numerous fundamental research questions that can only be successfully addressed when embracing methods from process metallurgy and physical metallurgy together. An example is the immense role of microstructure, fracture, and porosity on the direct reduction kinetics of solid oxide minerals exposed to hydrogen or hydrogen carriers [27].

Secondly, on more general grounds, there are multiple unexplored phenomena and basic research questions in the field of sustainable metallurgy [29]. This means that there is much room for new discoveries and the leverage of such discoveries on reducing the energy consumption and greenhouse gas emissions of these industries is potentially very large, equipping the urgent need for improved sustainability of the metallurgical sector with novel core competences. This becomes clear when recalling that about one-third of all industrial greenhouse gas emissions come from iron and steel making alone [29].

Common scientific motif

The common scientific motif behind the research activities in the department is the interplay between all the defects that make up an alloy’s microstructure and their local chemistry [93]. For revealing the underlying scientific laws of these interactions we conduct well-designed experiments and run them in concert with predictive simulations [5, 37, 67, 71, 108]. We use the resulting insights for engi-neering applications, particularly for inventing ad-vanced alloys and more metallurgical processes. More specific, the aim is the physics-based design of mate-rials with superior properties and sustainable process-es for the fields of energy, mobility, infrastructures, and health.

Material choices

We aim to understand the fundamental relationships between specific thermodynamic and kinetic features of materials and the evolution of microstructure and thus their effects on mechanical and functional properties. We therefore select, synthesize, process, and probe materials along specific intrinsic property gradients, such as their phase (meta-)stability, solid solution content, stacking fault energy, (athermal) transformation behaviour, magnetic hysteresis, or chemical reactivity. Along this systematics we work on the fundamentals of the relations between synthesis, microstructure, and properties of often complex and nanostructured materials. Focus is on metallic alloys such as aluminium, titanium, steels, high and medium entropy alloys, superalloys, magnesium, and magnetic and thermoelectric alloys.

Department structure and research groups

The department structure and organization reflects our understanding of fundamental yet highly flexible research. Some of the research groups pursue long-term visions of high strategic and methodological relevance for the department’s mission. Some other groups are non-permanent, and they are usually extramurally funded. This helps to rapidly establish new initiatives and to provide opportunities to young science leaders to pursue their own ideas and grow new initiatives (Fig. 3).

These non-permanent groups establish not only hubs for new research directions but they are also career vehicles for postdoctoral researchers who attract extramural funding, which allows them to establish own research initiatives. Funding agencies addressed for such initiatives are for instance the Max Planck -Fraunhofer collaboration scheme or the European Research Council, German Research Foundation, Federal Ministry of Education and Research (BMBF), and Volkswagen Foundation. These groups act as career platforms and rapid idea incubators, initiating new topics of highest novelty, quality, and scientific success. Most former group leaders of these initiatives have gained faculty positions at leading institutions around the globe.

Another category of groups is placed between the departments and between the MPIE and partner institutions elsewhere. These groups are often also funded by extramural third-party funds or by partnership programs of the Max Planck Society (MPG). They establish new

Fig. 3: *Research Groups in the Department Microstructure Physics and Alloy Design, together with Interdepartmental and Partner Groups.*

research fields which are placed between the individual departments and thus form centres for intense cross-disciplinary research, tapping competences from all departments and from centres outside of the institute. The interdepartmental and partner groups present their work in separate sections (see p. 23 - 35).

Long-term research groups

The long term research groups in the department work on Atom Probe Tomography (B. Gault), Computational Sustainable Metallurgy (J. Mianroodi), Mechanism-based Alloy Design (D. Ponge), Microscopy and Diffrac-tion (S. Zaefferer), and Theory and Simulation (F. Roters).

Atom Probe Tomography (B. Gault)

Atom Probe Tomography (APT) is a high-resolution characterization technique that provides three-dimensional elemental mapping with near-atomic resolution. Insights from APT help understand phase formation and transformations, segregation at interfaces, and partitioning between phases. The group focuses on applying this burgeoning microscopy and microanalysis technique to a wide range of advanced materials, with an emphasis on correlating the information gleaned from APT with other experimental and computational techniques [49, 101, 109–113]. The group has strong ties with numerous other groups within MPIE, in particular with the group for Mechanism-based Alloy Design, and across departments, with the Computational Phase Studies and the independent Nanoanalytics and Interfaces research group.

The group has operated since the installation of a local electrode atom probe Cameca LEAP 3000X HR in 2009. In August 2015, a second instrument, a LEAP 5000 XS was the first of its kind installed in the world, followed in February 2017 by a LEAP 5000 XR. The LEAP 3000 instrument was replaced in 2019 by another LEAP 5000 XR. These state-of-the-art instruments offer a wide field-of-view, high detection efficiency, and new generation of UV lasers. These instruments are complemented by two FEI Helios Dual Beam SEM/FIB (600/600i) and a FEI Helios PFIB.

The two LEAP 5000 XS and XR instruments are part of a world unique setup allowing for controlled transport of APT specimens from a glovebox to a scanning-electron microscope / focused ion beam to one of the two LEAPs via an ultra-high-vacuum suitcase. Transport of specimens can also be performed at cryogenic temperatures. This infrastructure was funded jointly by the BMBF and the MPG.

Computational Sustainable Metallurgy (J. Mianroodi)

The impact of humans on the environment and climate has long been scientific fact. Acceptance of this fact in the non-scientific world has finally reached the point where many societies and governments have started to adopt policy and to implement measures geared toward reduction or even elimination of this impact. Prominent examples of this include the idea of a climate-neutral, sustainable, and at least partially circular economy and production.

A number of scientific and technological challenges emerge from these considerations for the field of material engineering, metallurgy, steel production, energy storage, battery technology, and recycling. Solving these outstanding scientific challenges requires proper understanding of the underlying mechanisms and casting them into interdisciplinary, multi-physics and multi-scale modelling approaches. Advances in the understanding of chemical reactions, chemo-mechanical interaction, mechanical behaviour, defect evolution, and material degradation, from atomistic scales up to continuum level, are required to resolve many of the most pressing challenges and achieve the goal of material sustainability. These cover every aspect of material life from extraction to production, processing, manufacturing, service life, and recycling. This very recently established group thus focuses on applying and developing computational methods to solve various challenges related to materials and processes with important environmental impact, focusing on problems where the interplay of chemistry, phase transformation, microstructure, mechanics, and damage plays an important role enabling their deformation, mechanical response, and transport properties. Less well known is their influence on material chemistry. The severe lattice distortion at these defects drives solute segregation to them, resulting in strong, localized spatial variations in chemistry that determine microstructure and material behavior. Recent advances in atomic-scale characterization methods have made it possible to quantitatively resolve defect types and segregation chemistry. As shown here for a Pt-Au model alloy, we observe a wide range of defect-specific solute (Au[8, 11, 27, 39, 114, 115].

One example is the simulation of H-based direct reduction of iron ore as an alternative method of steel production with considerably lower emission compared to conventional carbon-based reduction for instance in blast furnaces. Modelling the reduction process by coupled chemo-mechanical phase-field methods including microstructure and defect evolution is an on-going research topic in this group. Close connection to the underlying physics at atomistic scales as well as experimental observation are used in the projects in this group.

Corrosion and damage evolution during the life cycle of metals is one of the most important underlying reason of component failure. Topics such as hydrogen embrittlement, stress corrosion cracking, Li-ion battery degradation are important for clean energy storage (either hydrogen storage or electricity) and are hence also in the focus of projects in this group. The interplay of chemistry, mechanics, and damage development at different scales is important in these topics.

Phase-field, finite element, molecular dynamics, Monte Carlo, and machine learning methods are the computational approaches used by the group. Software packages such as the Düsseldorf Advanced Material Simulation Kit (DAMASK), MOOSE framework, LAMMPS, and TensorFlow are among the tools employed.

Mechanism-based Alloy Design (D. Ponge)

This group works on the microstructure-oriented design of advanced high strength steels, high entropy alloys as well as on engineering Al-, Ni- and Ti-alloys (Fig. 4).

Projects focus on multiple strain hardening mechanisms such as the interplay of dislocations, twins, and deformation driven phase transformations. Of special interest are confined phase transformation phenomena at grain boundaries and dislocations [51, 79, 95]. Especially for materials with high strength, hydrogen embrittlement provides a challenge. Here the damage and failure mechanisms are analysed in order to develop novel high strength materials with low susceptibility against hydrogen embrittlement [13, 51, 85, 96, 97]. Projects in the

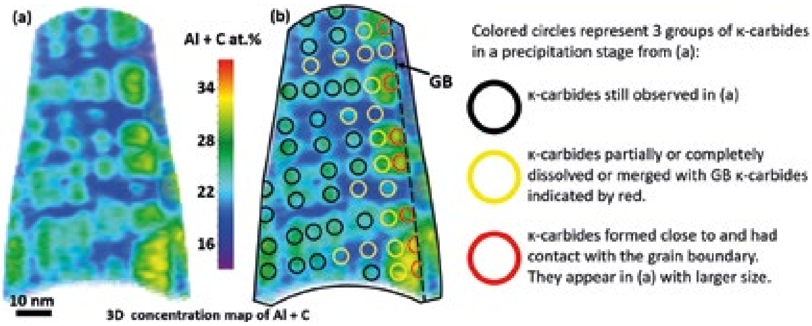


Fig. 4: *Formation and growth of grain boundary (GB) κ-carbides. a) 3D concentration map of Al+C of an APT sample containing a high angle grain boundary (GB) in an Fe-28Mn-8Al-1.3C wt.% alloy aged for 16 h at 550 °C. b) Overlaid image of (a) with a schematic of κ-carbides. κ-carbides formed by spinodal decomposition granting them the periodicity as indicated by coloured circles. The red group benefited from GB diffusion leading to faster growth. Their larger size allows them to consume the yellow group and grow to the sizes observed in (a). These nano-sized GB κ-carbides embrittle the GB in the presence of hydrogen or at low temperatures. During overaging these precipitates grow to micro-sizes and compromise the mechanical properties.*

group make intense use of the processing, mechanical testing, and microstructure characterization facilities at the institute down to the atomic scale. Projects are pursued in collaboration with partners from modelling, APT, and microscopy. Theory-guided thermomechanical processing is a main pathway for optimizing the microstructures and mechanical properties of complex alloys. In this context a main objective of projects in this group lies in understanding and utilizing elemental and mechanical partitioning effects among neighbouring phases on the one hand and among the matrix and lattice defects on the other hand with the aim to adjust the (meta-)stability of local phase states [6, 43, 98]. Depending on phase stability, deformation driven athermal transformations can be triggered such as spatially confined transformation-induced plasticity (TRIP) and transformation-inducted twinning (TWIP). Main examples are the design of ultrafine grained, partially metastable, maraging, multiphase, medium-manganese, martensite-to-austenite reversion, and weight reduced steels for automotive, manufacturing, and infrastructure applications.

Microscopy and Diffraction (S. Zaefferer)

This group works on two interconnected tasks: it aims at understanding microstructure formation mechanisms and the relation between microstructures and properties of materials by investigations on the microscopic level. To this aim the group develops or advances novel microscopy and diffraction techniques. Currently the focus lies on techniques in the scanning electron microscope, in particular on the electron diffraction techniques (EBSD, 3D EBSD, XR-EBSD, ECCI) [68, 71, 99–102]. Also, the group has expertise in the field of imaging and diffraction in transmission electron microscopy and X-ray diffraction. With respect to microstructure formation the group pursues questions on deformation, recrystallization, and solid-state phase transformations. Concerning microstructure-related properties the main focus is on mechanical properties, although, in recent years, also corrosion and electronic properties are studied. The spectrum of investigated materials comprises advanced high strength steels of 1st, 2nd, and 3rd generation (dual phase steels, low alloyed TRIP steels, complex phase steels, high-Mn steels, quench and partitioning steels), electrical steels, austenitic stainless steels, superalloys, magnesium-, aluminium- and copper alloys, intermetallic compounds (aluminides, Laves phases), and, since shortly, a range of photovoltaic materials (CdTe, Si, CIGS, GaAs).

Theory and Simulation (F. Roters)

This group develops constitutive models for advanced materials such as high strength steels. As the mechanical properties are of main interest crystal plasticity modelling builds the core of the activities. For this purpose, a number of constitutive models have been developed in the last 15 years. These models cover the full range from phenomenological descriptions to physics-based formulations of dislocation slip and other deformation mechanisms such as twinning induced plasticity (TWIP) and displacive transformations (TRIP). To facilitate the implementation of the models the Düsseldorf Advanced MAterial Simulation Kit (DAMASK, https://damask.mpie.de/) has been developed.

Meanwhile DAMASK was extended to a multi-field solver to enable treatment of multi-physics problems, e.g. thermal effects or damage. The full DAMASK capabilities together with many application examples have been published in an overview paper in 2019 [5]. The extended functionality necessitated a basic refracturing of the code together with a change of the in- and output formats. This work that culminated in the release of DAMASK 3 (see p. 73) was mainly performed in the new ICME group lead by M. Diehl (see p. 39).

One of the key challenges in using complex continuum scale constitutive models is the identification of (unique) parameter sets. To overcome this hurdle a new methodology has been developed to fit these parameters based on macroscopic properties [94]. The same procedures can be used to study parameter interdependencies and sensitivities, which in turn can help to improve the model formulation.

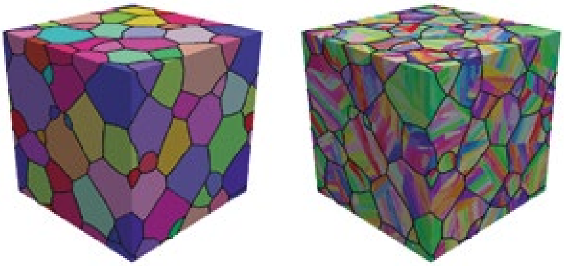


Fig. 5: *Creation of a martensitic representative volume element (RVE); synthetic austenitic microstructure (left) and resulting martensitic microstructure (right). Colours indicate different crystallographic orientations; black lines are grain boundaries.*

Besides reasonable parameter sets, successful microstructure-based simulation of properties relies on high quality representations of those microstructures. To this end a tool has been developed to create representative volume elements for martensite, Fig. 5 [95].

Many industrial processes involve large and heterogenous deformations. Simulation of such processes is challenging as heavily deformed meshes or grids often prevent convergence of the numerical algorithms used. The solution to this problem is remeshing/regridding to create new regular meshes or grids. While this is a standard technique in continuum mechanics, the transfer of microstructural state variables poses additional challenges. Two different strategies have been implemented in DAMASK to enable simulation of large deformations [96].

While all the above sounds rather technical it builds the foundation for the application of DAMASK to complex materials (e.g. bainitic steels) and processes (e.g. hot rolling including dynamic recrystallization) together with our industrial partners and in other groups within the MPIE.

Non-permanent research groups

Most of the more recently esablished non-permanent research groups are presented in separate sections, namely, High Performance Alloys for Extreme Environments (P. Kontis), the ERC-Shine group on Hydrogen in Materials (B. Gault) and Integrated Computational Materials Engineering (M. Diehl) (see p.39).

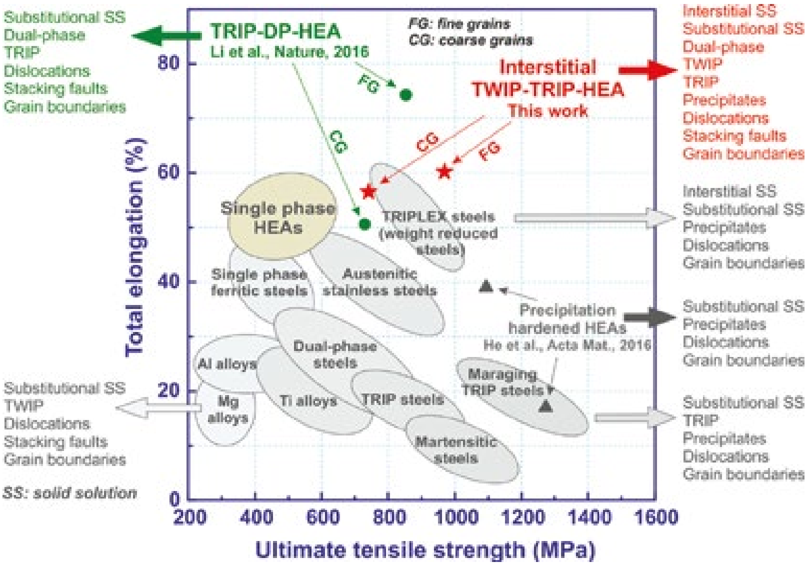


Fig. 6: *Strength-ductility profiles of various classes of metallic materials including novel HEAs recently developed by the group.*

Two of the non-permanent groups that were founded already before 2019 are the initiatives for High Entropy Alloys (Z. Li) and Materials Science of Mechanical Contacts (M. Herbig).

High Entropy Alloys (Z. Li)

The goal of this group is to develop novel high-entropy alloys (HEAs) with exceptional mechanical, physical and chemical properties based on the understanding of their structure-properties relations [21, 91, 106–110]. This is achieved by using advanced experimental techniques and the state-of-the-art theoretical methods, in close collaboration with the other groups and departments.

Conventional alloy design over the past centuries has been constrained by the concept of one or two prevalent base elements. As a breakthrough of this restriction, the concept of HEAs opens a new realm of numerous opportunities for investigations in the huge unexplored compositional space of multi-component alloys [111, 112].

While conventional alloys use strengthening mechanisms such as grain boundaries, dual-phase structure, dislocation interactions, precipitates, and solid solution (e.g., steels, Ti-alloys, Al-alloys), the novel interstitial TWIP-TRIP-HEAs concept developed by the group combines all available strengthening effects, namely, interstitial and substitutional solid solution, TWIP, TRIP, multiple phases, precipitates, dislocations, stacking faults, and grain boundaries [91, 108, 110, 113, 114]. This leads to the exceptional strength-ductility combination of the novel HEAs, exceeding that of most metallic materials (Fig. 6).

The group conducts projects along a few main topical fields associated with designing high-performance compositionally complex alloys, namely, materials with excellent strength-ductility combination, massive interstitial alloying, alloys with enhanced resistance to hydrogen-embrittlement and corrosion, and multifunctional materials including new alloys combining exceptional mechanical properties with specific magnetic and invar property profiles.

Materials Science of Mechanical Contacts (M. Herbig)

The group aims at deepening our understanding of the materials science phenomena associated with intense joint mechanical and environmental contacts [115–118]. This is key to revealing a broad range of phenomena required for improving crucial engineering components such as bearings, rails, hip implants, extrusion tools, boring heads, cutting inserts, or dental fillings.

The associated materials science of mechanical contacts is versatile and challenging (Fig. 7). The contact between two bodies subjected to high forces under harsh environmental conditions and multiple repetitions involves complex materials science phenomena: plastic deformation can lead to fatigue, grain refinement, and precipitate decomposition. Frictional heat can cause diffusion, phase transformation, recovery, or recrystallization. The presence of air, lubricants, or body fluids at the contact point causes oxidation, tribolayers, or even corrosion or hydrogen embrittlement. These processes usually occur simultaneously in service and cannot be tracked *in situ*. The analysis of such phenomena requires

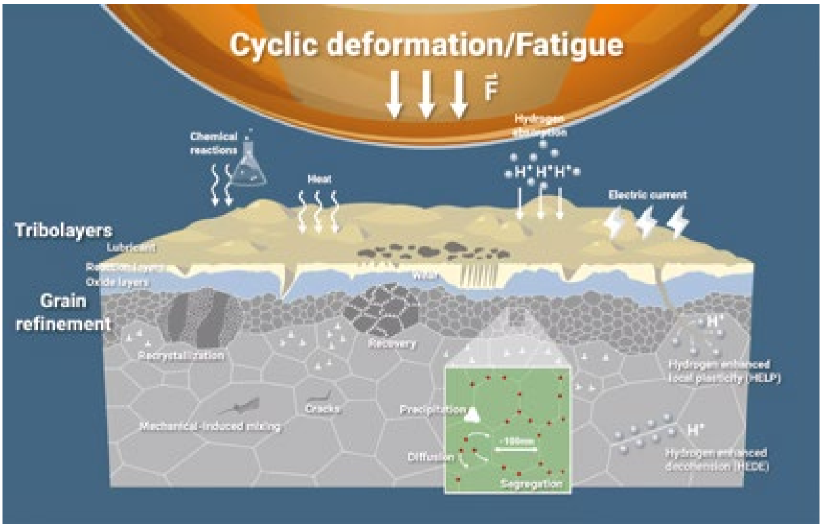


Fig. 7: *Microstructure reactions caused by intense mechanical/environmental contacts..*

combined chemical and structural characterization down to the atomic scale.

One main focus of the group are white-etching-cracks (WECs), which cause failure in bearings and rails, but which are in reality ubiquitous in high carbon steel applications subjected to intense mechanical contacts. This failure mode causes billions of euros costs worldwide each year. The group follows a staggered approach to yield a breakthrough on this long-standing challenge: Specimen failure under controlled laboratory conditions is generated using a customized rolling contact fatigue machine built in-house that simulates test conditions similar to ball bearings but on self-designed alloys. Both, lubrication and loading conditions as well as the electric current flow through the bearing (which is of importance as electric discharge events have been associated with the presence of WECs) can be controlled with this instrument. These specimens are compared to samples that failed during service using state-of-the-art microscopy. Individual phenomena are investigated separately where possible. Dedicated experiments are conducted to investigate the mechanisms of precipitate decomposition by deformation, heat, and electricity.

The research activities on steels are complemented by research on hip implants where corrosion and wear between the stem and head leads to adverse tissue reactions. Here, correlative transmission electron microscopy (TEM) and APT give access to the complex body/implant interactions.

The group uses all characterization techniques available at the MPIE, from rolling contact fatigue test rigs on whole bearings, over mesoscale investigations in the scanning electron microscope (SEM), down to the atomistic scale using TEM and APT. A key competence of the group is the use of correlative probing, for instance by combining these techniques to the same sample, to provide combined 3D structural and compositional information on near atomic scale, which is often the only way to answer a long-standing materials scientific question.

Grand challenges and recent breakthroughs

By bundling the core competences described above the department members jointly pursue a few 'Grand Challenge' topics, involving also members from all other departments. Details about some of these projects are given in the section "Interdepartmental Research Activities - Selected Highlights" (see. p. 182 - 225).

The 'Grand Challenges' we pursue are

1. Low-dimensional thermodynamics and kinetics at lattice defects
2. Understanding and solving hydrogen embrittlement
3. Compositionally complex alloys with special functional properties
4. Hydrogen-based reduction of iron oxides

Low-dimensional thermodynamics and kinetics at lattice defects

This topic is about the understanding and local chemi-cal manipulation of individual lattice defects. These features, such as dislocations and grain boundaries, influence mechanical, functional, and kinetic proper-ties of alloys. Such changes in the defect's chemical state can be predicted and experimentally studied under thermodynamic well-controlled grand canonical conditions, for instance by single or multi-component solute decoration. This change in their chemical state leads to changes in energy, mobility, structure, cohe-sion and can even promote local phase transformation. In an approach referred to as 'segregation engineering' such solute decoration is not

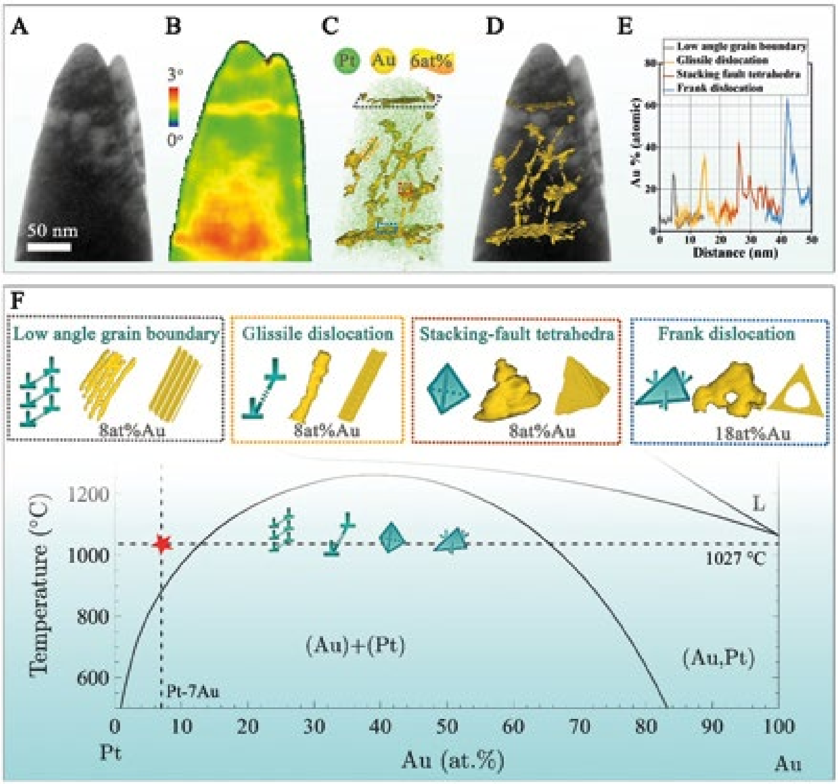


Fig. 8: *Study of a Pt–7 at % Au thin film annealed at 1300 K for 15 min.(A) TEM-based bright-field image of specimen tip oriented normal to the (-1 1 -1) crystallographic direction. (B) Misorientation map from electron diffraction. (C) APT-based reconstruction of the 6 at % segregated Au isosurface superimposed on the bright-field image from (A). The x at % isosurface represents the region (voxels) containing x or more at % Au. (D) Spatial distribution of Pt (green) and Au (gold) atoms in the specimen tip and the isosurfaces from (C). (E) Au at % profiles across particular defects shown in the upper panels in (F). The direction of the profiles in (E) is indicated by the arrows in (F). These include (i) Low-angle grain boundary (grey profile normal to dislocation array), (ii) glissile dislocation (yellow profile parallel to slip plane normal), (iii) stacking fault tetrahedron, and (iv) Frank loop. (F) Top: Each panel shows an image (middle) of each observed defect type, corresponding defect symbols (left), and a DMD simulation result (right) in the coloured boxes. The numbers in each box are the Au at % isosurface (Iso) values for imaging (left) and maximum (max) solute at % near the defects (right). The maximum values are obtained from the Au at % profile in (E). A length of 5 nm is indicated by the bar. Bottom: equilibrium Pt-Au phase diagram [11].*

regarded as an undesired phenomenon but it is instead utilized to manipulate specific defect structures and properties via local composition tuning [57, 119, 137–139].

As an example, for the case of one-dimensional defects we studied the solute decoration of dislocations. Dislocations are one-dimensional defects in crystals, enabling their deformation, mechanical response, and transport properties. Less well-known is their influence on material chemistry. The severe lattice distortion at these defects drives solute segregation to them, resulting in strong, localized spatial variations in chemistry, that determine microstructure and material behaviour. Recent advances in high resolution correlative atomic-scale material characterization have made it possible to quantitatively resolve different dislocation types and arrangements and their respective segregation chemistry. One example for a Pt-Au model alloy is shown in Fig. 8 [11]. We observe here a wide range of defect-specific solute (Au) decoration patterns of much greater variety and complexity than expected from the traditional Cottrell cloud picture. The solute decoration of the dislocations can be up to half an order of magnitude higher than expected from classical theory, and the differences are determined by their structure, mutual alignment, and distortion field. This opens up pathways to utilize dislocations for the compositional and structural nanoscale design of advanced materials. We made similar observations also for Fe-Mn and Al-Mg alloys, revealing by combined atomic-scale characterization and simulation the complexity and diversity of the chemical nature of dislocations.

We studied similar phenomena for the case of grain boundary segregation and found for example complex planar compositional decomposition patterns and even local austenite reversion of these decorated interface regions. In an Fe-9 wt.% Mn martensite we studied grain boundary embrittlement in the quenched and tempered state. While solute Mn directly embrittles martensite grain boundaries, reversion of martensite back to austenite at grain boundaries cleans the interfaces from solute Mn by partitioning the Mn into the newly formed austenite, hence restoring impact toughness and fatigue resistance. The grain boundary segregation is a local equilibrium state and shifts the chemical working point from the material's bulk composition to a locally much higher composition. With this shift in the local concentration a number of spatially-confined (hence low-dimensional) thermodynamic phenomena can be observed at lattice defects that do not occur in the adjacent bulk regions.

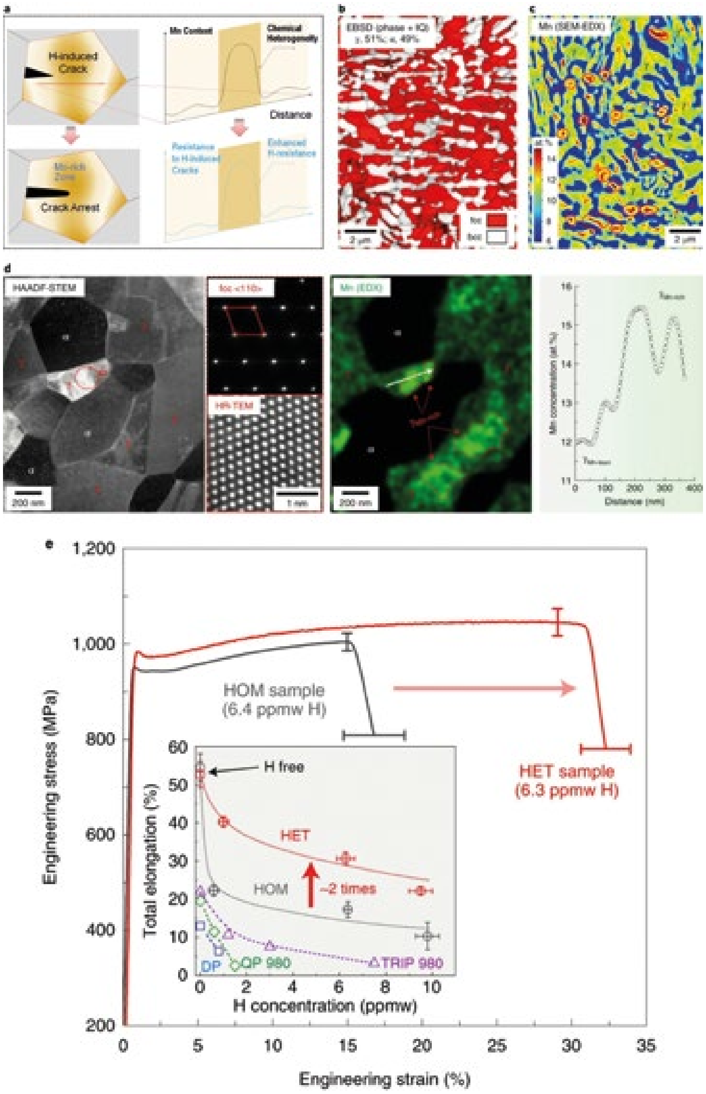


Fig. 9: *a, Schematic image of the concept, showing a H-induced crack propagation crossing a designed solute-rich buffer region, with the solute concentration profile and the corresponding crack resistance schematically shown on the right side. b, Electron backscatter diffraction (EBSD) phase plus image quality (IQ) map showing the austenite–ferrite dual-phase microstructure. fcc, face-centred cubic; bcc, body-centred cubic. c, Scanning electron microscopy (SEM)-based energy-dispersive X-ray spectroscopy (EDX) map revealing the overall Mn distribution pattern in the microstructure. The chemical buffer zones are those regions where Mn is highly enriched (~14–16 at. % Mn) inside the austenite phase (some of them are marked by elliptical frames). d, High-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) observation with EDX analysis, showing the existence of multiple Mn-rich zones inside one austenite crystal cluster or even one austenite grain. The selected area electron diffraction and high-resolution transmission electron microscopy (HR-TEM) images taken, respectively, from the marked circular and rectangular frames are placed on the right side of the STEM image. The EDX line profile is taken from the area marked by a white arrow in the EDX map. e, Mechanical properties of the chemically homogeneous (HOM) and of the chemically heterogeneous (HET) alloy variant [95].*

Understanding and solving hydrogen embrittlement

One of the main challenges in the field of hydrogen embrittlement is to understand the relationships between the individual hydrogen-induced mechanisms and their synergistic interplay in creating actual damage initiation features that can lead to catastrophic material failure.

Some of the individual effects that hydrogen causes when intruding metallic alloys are in part well-established. An example is the hydrogen enhanced local plasticity (HELP) effect in metals with BCC lattice structure such as ferritic steels. This leads to the local softening of the material due to enhanced mobility of the dislocations. Another effect is the enhanced decohesion across interfaces that can be caused by hydrogen decoration (HEDE). Another important effect is the stabilization of vacancies by hydrogen decoration, leading to super-abundant vacancy concentrations which can condensate and lead to porosity. What is much less understood, however, is how these individual mechanisms actually act together to cause catastrophic damage and failure, Fig. 9 [95].

In this context, measurements including sufficient spatial resolution of the associated phenomena down to the lattice defect scale are still hard to realize. Yet, the role of individual lattice defects and surface features together with a characterization of their individual structural and chemical state is essential for understanding corrosion and the resulting decay and failure mechanisms. Specifically, site-specific microstructure and composition characterization is of great relevance. Instrumentation we use in this context are correlative atom probe tomography, Scanning Kelvin Probe mapping (together with the departments of G. Dehm and M. Rohwerder) in conjunction with EBSD and ECCI as well as nanoscale secondary ion mass spectrometry (nanoSIMS). In the context of imaging hydrogen and deuterium at the atomic scale particularly the new atom probe laboratory which is equipped with a cryo-transfer unit and a charging glove box is essential. Its use allows charged samples to be exchanged and probed among different microscopes and atom probes without any exposure to air or elevated temperatures (see p. 50). More details on the latest instrumental progress at the MPIE are presented on pages see p. 50 and p. 204.

These spatially resolved hydrogen mapping methods enabled us not only to establish a relationship between localised plasticity and cohesion effects in steels but also to develop a microstructure design approach that substantially enhances the material's resistance against hydrogen embrittlement. More specific we used our findings to introduce a counterintuitive strategy where we exploit typically undesired chemical heterogeneity within the material's microstructure that enables the local enhancement of crack resistance and local hydrogen trapping (Fig. 9). We deploy this approach to a manganese-containing high-strength steel and produce a high dispersion of manganese-rich zones in the microstructure. These solute-rich buffer regions allow for local micro-tuning of the phase stability, arresting H-induced microcracks thus interrupting the hydrogen-assisted damage percolation. This effect results in a superior hydrogen embrittlement resistance (by a factor of two) without sacrificing the material's strength and ductility. The strategy of exploiting chemical heterogeneities, rather than avoiding them, broadens the horizon for microstructure engineering via advanced thermomechanical processing.

Compositionally complex alloys with special functional properties

An important class of compositionally complex alloys with a wide range of mutual solid solubility has been identified for mixtures of the elements Fe, Ni, Co, Cr and Mn. These elements offer not just interesting opportunities for tuning the stacking fault energy and enhancing the solid solution strength, but they are also known for their strong spin coupling: Fe, Ni and Co are the strongest single element ferromagnets while Cr and Mn are the strongest antiferromagnets. These features, together with a wide range of adjustable valence electron concentrations, has triggered interest in developing alloys with good magnetic and/or invar properties (Fig. 10).

One project in that context targets mechanically strong soft-magnetic alloys. The motivation behind that is that a lack of strength and damage tolerance can limit the applications of conventional soft magnetic materials, particularly in mechanically loaded functional devices which are critical for the electrification of transport and industry. Therefore, strengthening of these materials is important. However, conventional strengthening concepts usually significantly deteriorate soft magnetic properties, due to Bloch wall interactions with the defects used for hardening. We followed therefore a novel concept to overcome this dilemma, by developing bulk alloys with excellent mechanical and attractive soft magnetic properties through coherent and ordered nanoprecipitates (<15 nm) dispersed homogeneously within a face-centred cubic matrix of a non-equiatomic CoFeNiTaAl high-entropy alloy (HEA) [54]. Compared to the alloy in precipitate-free state, the alloy variant with a large volume fraction (>42 %) of nanoprecipitates achieves significantly enhanced strength (≈1526 MPa) at good ductility (≈15 %), while the coercivity is only marginally increased (<10.7 Oe). The ordered nanoprecipitates and the resulting dynamic microband refinement in the matrix significantly strengthen the HEAs, while full coherency between the nanoprecipitates and the matrix leads at the same time to the desired insignificant pinning of the magnetic domain walls.

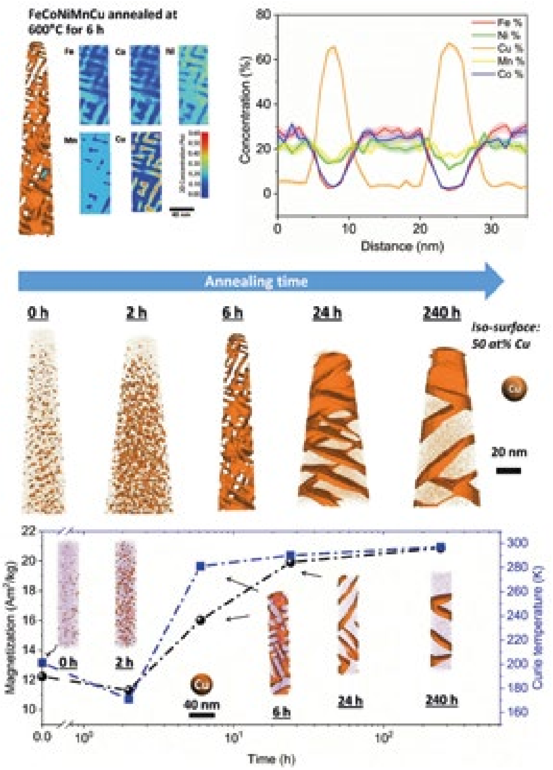


Fig. 10: *APT and magnetic analysis of a homogenized* $Fe_{15}Co_{15}Ni_{20}Mn_{20}Cu_{30}$ *high entropy alloy annealed at 600 °C for several times. APT tip reconstructions of Fe, Ni, Co, Mn, and Cu. 50 at. % iso-concentration surfaces of Cu and in-plane compositional analysis of the identical sample region marked on the isoconcentration surfaces [89, 106].*

Another approach made use of tailoring magnetic properties via spinodal decomposition in compositionally complex alloys, Fig. 10 [89, 106]. While the original version of the high-entropy alloy concept aimed at stabilizing single- or dual-phase multi-element solid solutions through high mixing entropy, we modified this strategy by rendering such massive solid solutions metastable, to trigger spinodal decomposition for improving the alloys' magnetic properties. The motivation for starting from a complex composition for this approach is to provide the chemical degrees of freedom required to tailor spinodal behaviour using multiple components. The key idea is to form Fe-Co enriched regions which have an expanded volume (relative to unconstrained Fe-Co), due to coherency constraints imposed by the surrounding HEA matrix. As demonstrated by theory and experiments, this leads to improved magnetic properties of the decomposed alloy relative to the original solid solution matrix. In a prototype magnetic FeCoNiMnCu HEA, we could show that the modulated structures, achieved by spinodal decomposition, lead to an increase of the Curie temperature by 48 % and a simultaneous increase of magnetization by 70 % at ambient temperature as compared to the homogenized single-phase reference alloy. The findings thus open a pathway for the development of advanced functional compositionally complex alloys.

Hydrogen-based reduction of iron oxides

Steel is the most important material class in terms of volume and environmental impact. While it is a sustainability enabler, for instance through lightweight design, magnetic devices, and efficient turbines, its primary production is not. Iron is reduced from ores by carbon, causing 30 % of the global CO_2 emissions in manufacturing, qualifying it as the largest single industrial greenhouse gas emission source. Hydrogen is thus attractive as alternative reductant. Although this reaction has been studied for decades, its kinetics is not well understood, particularly during the wüstite reduction step which is much slower than hematite reduction.

Some rate-limiting factors of this reaction are determined by the microstructure and local chemistry of the ores. Here, we report on a multi-scale structure and composition analysis of iron reduced from hematite with pure H_2, reaching down to near-atomic scale. During reduction a complex pore- and microstructure evolves, due to oxygen loss and non-volume conserving phase transformations. The microstructure after reduction is an aggregate of nearly pure iron crystals, containing inherited and acquired pores and cracks. We observe several types of lattice defects that accelerate mass transport as well as several chemical impurities (Na, Mg, Ti, V) within the Fe in the form of oxide islands that were not reduced. With this work, we aim to open the perspective in the field of carbon-neutral iron H-based direct reduction from macroscopic processing towards better understanding of the underlying microscopic transport and reduction mechanisms and kinetics [27].

Reduction based on a hydrogen plasma may offer an attractive alternative to direct reduction. We therefore also study the reduction of hematite using hydrogen plasma. The evolution of both, chemical composition and phase transformations was investigated in several intermediate states. We found that hematite reduction kinetics depends on the balance between the initial input mass and the arc power. For an optimized input mass-arc power ratio, complete reduction was obtained within 15 min of exposure to the hydrogen plasma. In such a process, the wüstite reduction is also the rate-limiting step for complete reduction. Nonetheless, the reduction reaction is exothermic, and its rates are comparable with those found in hydrogen-based direct reduction. Micro- and nanoscale chemical and microstructure analysis revealed that the gangue elements partition to the remaining oxide regions, probed by energy dispersive spectroscopy (EDS) and APT. Si-enrichment was observed in the interdendritic fayalite domains, at the wüstite/iron hetero-interfaces and in the primarily solidified oxide particles inside the iron. With proceeding reduction, however, such elements are gradually removed from the samples so that the final iron product is nearly free of gangue-related impurities. Our findings provide microstructural and atomic-scale insights into the composition and phase transformations occurring during iron ore reduction by hydrogen plasma, propelling better understanding of the underlying thermodynamics and kinetic barriers of this essential process [28].

References

1. Morgado, F. F.; Katnagallu, S.; Freysoldt, C.; Klaes, B.; Vurpillot, F.; Neugebauer, J.; Raabe, D.; Neumeier, S.; Gault, B.; Stephenson, L. T.: Scr. Mater. 203 (2021) 114036.
2. Katnagallu, S.; Nematollahi, A.; Dagan, M.; Moody, M.; Grabowski, B.; Gault, B.; Raabe, D.; Neugebauer, J.: Microsc. Microanal. 23 (2017) 642.
3. Katnagallu, S.; Gault, B.; Grabowski, B.; Neugebauer, J.; Raabe, D.; Nematollahi, A.: Mater. Charact. 146 (2018) 307.
4. Fan, H.; Wang, Q.; El-Awady, J. A.; Raabe, D.; Zaiser, M.: Nat. Commun. 12 (2021) 1.
5. Roters, F.; Diehl, M.; Shanthraj, P.; Eisenlohr, P.; Reuber, C.; Wong, S. L.; Maiti, T.; Ebrahimi, A.; Hochrainer, T.; Fabritius, H.-O.; Nikolov, S.; Friák, M.; Fujitai, N.; Grilli, N.; Janssens, K. G. F.; Jia, N.; Kok, P. J. J.; Ma, D.; Meier, F.; Werner, E.; Stricker, M.; Weygand, D.; Raabe, D.: Comput. Mater. Sci. 158 (2019) 420.
6. Souza Filho, I. R.; Sandim, M. J. R. J. R.; Ponge, D.; Sandim, H. R. Z. R. Z.; Raabe, D.: Mater. Sci. Eng. A 754 (2019) 636.
7. Sulzer, S.; Li, Z.; Zaefferer, S.; Hafez Haghighat, S. M.; Wilkinson, A.; Raabe, D.; Reed, R.: Acta Mater. 185 (2020) 13.
8. Wu, X.; Makineni, S. K.; Liebscher, C. H.; Dehm, G.; Rezaei Mianroodi, J.; Shanthraj, P.; Svendsen, B.; Bürger, D.; Eggeler, G.; Raabe, D.; Gault, B.: Nat. Commun. 11 (2020) 1.
9. Wu, X.; Makineni, S. K.; Kontis, P.; Dehm, G.; Raabe, D.; Gault, B.; Eggeler, G.: Materialia 4 (2018) 109.
10. Benzing, J. T.; Kwiatkowski da Silva, A.; Morsdorf, L.; Bentley, J.; Ponge, D.; Dutta, A.; Han, J.; McBride, J. R.; Van Leer, B.; Gault, B.; Raabe, D.; Wittig, J. E.: Acta Mater. 166 (2019) 512.
11. Zhou, X.; Mianroodi, J. R.; Da Silva, A. K.; Koenig, T.; Thompson, G. B.; Shanthraj, P.; Ponge, D.; Gault, B.; Svendsen, B.; Raabe, D.: Sci. Adv. 7 (2021) 1.
12. Kwiatkowski Da Silva, A.; Ponge, D.; Peng, Z.; Inden, G.; Lu, Y.; Breen, A.; Gault, B.; Raabe, D.: Nat. Commun. 9 (2018) 1.
13. Raabe, D.; Sun, B.; Kwiatkowski Da Silva, A.; Gault, B.; Yen, H. W.; Sedighiani, K.; Thoudden Sukumar, P.; Souza Filho, I. R.; Katnagallu, S.; Jägle, E.; Kürnsteiner, P.; Kusampudi, N.; Stephenson, L.; Herbig, M.; Liebscher, C.H.; Springer, H.; Zaefferer, S.; Shah, V.; Wong, S. L. ; Baron, C. ; Diehl, M.; Roters, F.; Ponge, D.: Mater. Trans. A Phys. Metall. Mater. Sci. 51 (2020) 5517.
14. Koyama, M.; Zhang, Z.; Wang, M.; Ponge, D.; Raabe, D.; Tsuzaki, K.; Noguchi, H.; Tasan, C. C.: Science 355 (2017) 1055.

15. Ding, R.; Yao, Y.; Sun, B.; Liu, G.; He, J.; Li, T.; Wan, X.; Dai, Z.; Ponge, D.; Raabe, D.; Zhang, C.; Godfrey, A.; Miyamoto, G.; Furuhara, T.; Yang, Z.; van der Zwaag, S.; Chen, H.: Sci. Adv. 6 (2020) eaay1430.
16. Lei, Z.; Wu, Y.; He, J.; Liu, X.; Wang, H.; Jiang, S.; Gu, L.; Zhang, Q.; Gault, B.; Raabe, D.; Lu, Z.: Sci. Adv. 6 (2020) eaba7802.
17. Springer, H.; Zhang, J. L.; Szczepaniak, A.; Belde, M.; Gault, B.; Raabe, D.: Sci. Eng. A 762 (2019) 138088.
18. Jiang, S.; Wang, H.; Wu, Y.; Liu, X.; Chen, H.; Yao, M.; Gault, B.; Ponge, D.; Raabe, D.; Hirata, A. Chen, M.; Wang, Y.; Lu, Z.: Nature 544 (2017) 460.
19. Song, R.; Ponge, D.; Raabe, D.; Speer, J. G.; Matlock, D. K.: Mater. Sci. Eng. A. 441 (2006) 1.
20. Han, J.; Kang, S. H.; Lee, S. J.; Kawasaki, M.; Lee, H. J.; Ponge, D.; Raabe, D.; Lee, Y. K.: Nat. Commun. 8 (2017) 8.
21. Li, Z.; Ludwig, A.; Savan, A.; Springer, H.; Raabe, D.: J. Mater. Res. 33 (2018) 3156.
22. Raabe, D.; Roters, F.; Neugebauer, J.; Gutierrez-Urrutia, I.; Hickel, T.; Bleck, W.; Schneider, J. M.; Wittig, J. E.; Mayer, J.: MRS Bull. 41 (2016) 320.
23. Knoll, H.; Ocylok, S.; Weisheit, A.; Springer, H.; Jägle, E.; Raabe, D.: Steel Res. Int. 88 (2017) 1.
24. Schnabel, V.; Köhler, M.; Evertz, S.; Gamcova, J.; Bednarcik, J.; Music, D.; Raabe, D.; Schneider, J. M.: Acta Mater. 107 (2016) 213.
25. Raabe, D.; Springer, H.; Gutierrez-Urrutia, I.; Roters, F.; Bausch, M.; Seol, J. B.; Koyama, M.; Choi, P. P.; Tsuzaki, K.: JOM 66 (2014) 1845.
26. Luo, H.; Sohn, S. S.; Lu, W.; Li, L.; Li, X.; Soundararajan, C. K.; Krieger, W.; Li, Z.; Raabe, D.: Nat. Commun. 11 (2020) 1.
27. Kim, S. H.; Zhang, X.; Ma, Y.; Souza Filho, I. R.; Schweinar, K.; Angenendt, K.; Vogel, D.; Stephenson, L. T.; El-Zoka, A. A.; Mianroodi, J. R.; Rohwerder, M.; Gault, B.; Raabe, D.: Acta Mater. 212 (2021) 116933.
28. Souza Filho, I. R.; Ma, Y.; Kulse, M.; Ponge, D.; Gault, B.; Springer, H.; Raabe, D.: Acta Mater. 213 (2021) 116971.
29. Raabe, D.; Tasan, C. C.; Olivetti, E. A.: Nature 575 (2019) 64.
30. Kürnsteiner, P.; Bajaj, P.; Gupta, A.; Wilms, M. B.; Weisheit, A.; Li, X.; Leinenbach, C.; Gault, B.; Jägle, E. A.; Raabe, D.: Addit. Manuf. 32 (2020) 100910.
31. Chauvet, E.; Kontis, P.; Jägle, E. A.; Gault, B.; Raabe, D.; Tassin, C.; Blandin, J.-J. J.; Dendievel, R.; Vayre, B.; Abed, S.; Martin, G.: Acta Mater. 142 (2018) 82.
32. Jägle, E. A.; Sheng, Z.; Wu, L.; Lu, L.; Risse, J.; Weisheit, A.; Raabe, D.: JOM 68 (2016) 943.
33. Kürnsteiner, P.; Wilms, M. B.; Weisheit, A.; Barriobero-Vila, P.; Jägle, E. A.; Raabe, D.: Acta Mater. 129 (2017) 52.
34. 34, Bajaj, P.; Hariharan, A.; Kini, A.; Kürnsteiner, P.; Raabe, D.; Jägle, E. A.: Mater. Sci. Eng. A 772 (2020) 138633.
35. Makineni, S. K.; Kini, A. R.; Jägle, E. A.; Springer, H.; Raabe, D.; Gault, B.: Acta Mater. 151 (2018) 31.
36. Kürnsteiner, P.; Wilms, B. M.; Weisheit, A.; Gault, B.; Jägle, E. A.; Raabe, D.: Nature 582 (2020) 515.
37. Sedighiani, K.; Diehl, M.; Traka, K.; Roters, F.; Sietsma, J.; Raabe, D.: Int. J. Plast. 134 (2020) 102779.
38. Han, F.; Diehl, M.; Roters, F.; Raabe, D.: J. Mater. Process. Technol. 277 (2020) 116449.
39. Diehl, M.; Wang, D.; Liu, C.; Rezaei Mianroodi, J.; Han, F.; Ma, D.; Kok, P. J. J.; Roters, F.; Shanthraj, P.: Adv. Eng. Mater. 3 (2020) 1901044.
40. Wei, Y.; Gault, B.; Varanasi, R. S.; Raabe, D.; Herbig, M.; Breen, A. J.: Ultramicroscopy 194 (2018) 15.
41. Wei, Y.; Varanasi, R. S.; Schwarz, T.; Gomell, L.; Zhao, H.; Larson, D. J.; Sun, B.; Liu, G.; Chen, H.; Raabe, D.: Patterns 2 (2021) 100192.
42. Mianroodi, J. R.; H. Siboni, N.; Raabe, D.: npj Comput. Mater. 7 (2021) 1.
43. Kwiatkowski da Silva, A.; Leyson, G.; Kuzmina, M.; Ponge, D.; Herbig, M.; Sandlöbes, S.; Gault, B.; Neugebauer, J.; Raabe, D.: Acta Mater. 124 (2017) 305.
44. Garner, A.; Euesden, R.; Yao, Y.; Aboura, Y.; Zhao, H.; Donoghue, J.; Curioni, M.; Gault, B.; Shanthraj, P.; Barrett, Z.; Engel, C.; Burnett, T. L.; Prangnell, P. B.: Acta Mater. 202 (2020) 190.
45. Chang, Y. H.; Mouton, I.; Stephenson, L.; Ashton, M.; Zhang, G. K.; Szczpaniak, A.; Lu, W. J.; Ponge, D.; Raabe, D.; Gault, B.: New J. Phys. 21 (2019) 53025.
46. Chang, Y.; Lu, W.; Guénolé, J.; Stephenson, L. T.; Szczpaniak, A.; Kontis, P.; Ackerman, A. K.; Dear, F. F.; Mouton, I.; Zhong, X.; Zhang, S.; Dye, D.; Liebscher, C. H.; Ponge, D., Korte-Kerzel, S.; Raabe, D.; Gault, B.: Nat. Commun. 10 (2019) 942.
47. Li, Z.; Tasan, C. C.; Springer, H.; Gault, B.; Raabe, D.: Sci. Rep. 7 (2017) 1.
48. Kovács, A.; Lewis, L. H.; Palanisamy, D.; Denneulin, T.; Schwedt, A.; Scott, E. R. D.; Gault, B.; Raabe, D.; Dunin-Borkowski, R. E.; Charilaou, M.: Nano Lett. 21 (2021) 8135.
49. Makineni, S. K.; Lenz, M.; Kontis, P.; Li, Z.; Kumar, A.; Felfer, P. J.; Neumeier, S.; Herbig, M.; Spiecker, E.; Raabe, D.; Gault, B.: JOM 70 (2018) 1736.
50. George, E. P.; Raabe, D.; Ritchie, R. O.: Nat. Rev. Mater. 4 (2019) 515.
51. Sun, B.; Palanisamy, D.; Ponge, D.; Gault, B.; Fazeli, F.; Scott, C.; Yue, S.; Raabe, D.: Acta Mater. 164 (2019) 683.
52. Seol, J. B.; Na, S. H.; Gault, B.; Kim, J. E.; Han, J. C.; Park, C. G.; Raabe, D.: Sci. Rep. 7 (2017) 1.
53. Li, Z.; Körmann, F.; Grabowski, B.; Neugebauer, J.; Raabe, D.: Acta Mater. 136 (2017) 262.
54. Han, L.; Rao, Z.; Souza Filho, I. R.; Maccari, F.; Wei, Y.; Wu, G.; Ahmadian, A.; Zhou, X.; Gutfleisch, O.; Ponge, D.; Raabe, D.; Li, Z.: Adv. Mater. 33 (2021) 202102139.
55. Abdellaoui, L.; Zhang, S.; Zaefferer, S.; Bueno-Villoro, R.; Baranovskiy, A.; Cojocaru-Mirédin, O.; Yu, Y.; Amouyal, Y.; Raabe, D.; Snyder, G. J.; Scheu, C.: Acta Mater. 178 (2019) 135.
56. Abdellaoui, L.; Chen, Z.; Yu, Y.; Luo, T.; Hanus, R.; Schwarz, T.; Bueno Villoro, R.; Cojocaru Mirédin, O.; Snyder, G. J.; Raabe, D.: Adv. Funct. Mater. 31 (2021) 2101214.
57. Yu, Y.; Zhou, C.; Zhang, S.; Zhu, M.; Wuttig, M.; Scheu, C.; Raabe, D.; Snyder, G. J.; Gault, B.; Cojocaru-Mirédin, O.: Mater. Today 32 (2020) 260.
58. Luo, T.; Serrano-Sánchez, F.; Bishara, H.; Zhang, S.; Villoro, B.; Kuo, J. J.; Felser, C.; Scheu, C.; Snyder, G. J.; Best, J. P.; Dehm, G.; Yu, Y.; Raabe, D.; Fu, C.; Gault, B.: Acta Mater. 217 (2021) 117147.
59. Cojocaru-Mirédin, O.; Abdellaoui, L.; Nagli, M.; Zhang, S.; Yu, Y.; Scheu, C.; Raabe, D.; Wuttig, M.; Amouyal, Y.: ACS Appl. Mater. Interfaces 9 (2017) 14779.
60. Raabe, D.; Li, Z.; Ponge, D.: MRS Bull. 44 (2019) 266.

61. Benzing, J. T.; Bentley, J.; McBride, J. R.; Ponge, D.; Han, J.; Raabe, D.; Wittig, J. E.: Microsc. Microanal. 23 (2017) 402.
62. Han, J.; da Silva, A. K.; Ponge, D.; Raabe, D.; Lee, S. M.; Lee, Y. K.; Lee, S. I.; Hwang, B.: Acta Mater. 122 (2017) 199.
63. Dutta, A.; Ponge, D.; Sandlöbes, S.; Raabe, D.: Materialia 5 (2019) 100252.
64. Palanisamy, D.; Ener, S.; Maccari, F.; Schäfer, L.; Skokov, K. P.; Gutfleisch, O.; Raabe, D.; Gault, B.: Phys. Rev. Mater. 4 (2020) 54404.
65. Guillon, O.; Elsässer, C.; Gutfleisch, O.; Janek, J.; Korte-Kerzel, S.; Raabe, D.; Volkert, C. A.: Mater. today 21 (2018) 527.
66. Schönhöbel, A. M.; Madugundo, R.; Barandiarán, J. M.; Hadjipanayis, G. C.; Palanisamy, D.; Schwarz, T.; Gault, B.; Raabe, D.; Skokov, K.; Gutfleisch, O.; Fischbacher, J.; Schrefl, T.: Acta Mater. 200 (2020) 652.
67. Diehl, M.; Wicke, M.; Shanthraj, P.; Roters, F.; Brueckner-Foit, A.; Raabe, D.: JOM 69 (2017) 872.
68. Shanthraj, P.; Diehl, M.; Eisenlohr, P.; Roters, F.; Raabe, D.; Chen, C. S.; Chawla, K. K.; Chawla, N.; Chen, W.; Kagawa, Y.: Handb. Mech. Mater. Springer, Berlin (2019) 978.
69. Svendsen, B.; Shanthraj, P.; Raabe, D.: J. Mech. Phys. Solids 112 (2018) 619.
70. Liu, C.; Garner, A.; Zhao, H.; Prangnell, P. B.; Gault, B.; Raabe, D.; Shanthraj, P.: Acta Mater. 214 (2021) 116966.
71. Liu, C.; Shanthraj, P.; Diehl, M.; Roters, F.; Dong, S.; Dong, J.; Ding, W.; Raabe, D.: Int. J. Plast. 106 (2018) 203.
72. Diehl, M.; Shanthraj, P.; Eisenlohr, P.; Roters, F.: Meccanica 51 (2016) 429.
73. Han, F.; Roters, F.; Raabe, D.: Int. J. Plast. 125 (2020) 97.
74. Darvishi Kamachali, R.; Schwarze, C.; Lin, M.; Diehl, M.; Shanthraj, P.; Prahl, U.; Steinbach, I.; Raabe, D.: Comput. Mater. Sci. 155 (2018) 541.
75. Gault, B.; Chiaramonti, A.; Cojocaru-Mirédin, O.; Stender, P.; Dubosq, R.; Freysoldt, C.; Makineni, S. K.; Li, T.; Moody, M.; Cairney, J. M.: Nat. Rev. Methods Prim. 1 (2021) 51.
76. Kühbach, M.; Bajaj, P.; Zhao, H.; Çelik, M. H.; Jägle, E. A.; Gault, B.: npj Comput. Mater. 7 (2021) 1.
77. Morales-Rivas, L.; Archie, F.; Zaefferer, S.; Benito-Alfonso, M.; Tsai, S.-P. P.; Yang, J.-R. R.; Raabe, D.; Garcia-Mateo, C.; Caballero, F. G.: J. Alloys Compd. 752 (2018) 505.
78. Breitbarth, E.; Zaefferer, S.; Archie, F.; Besel, M.; Raabe, D.; Requena, G.: Mater. Sci. Eng. A 718 (2018) 345.
79. Godec, M.; Zaefferer, S.; Podgornik, B.; Šinko, M.; Tchernychova, E.: Mater. Charact. 160 (2020) 1583.
80. Zaefferer, S.; Elhami, N. N.: Acta Mater. 75 (2014) 20.
81. Konijnenberg, P.; Khorashadizadeh, A.; Zaefferer, S.; Raabe, D.: Microsc. Microanal. 19 (2013) 846.
82. Stechmann, G.; Zaefferer, S.; Konijnenberg, P.; Raabe, D.; Gretener, C.; Kranz, L.; Perrenoud, J.; Buecheler, S.; Tiwari, A. N.: Sol. Energy Mater. Sol. Cells 151 (2016) 66.
83. Konijnenberg, P. J.; Zaefferer, S.; Raabe, D.: Acta Mater. 99 (2015) 402.
84. An, D.; Griffiths, T. A.; Konijnenberg, P.; Mandal, S.; Wang, Z.; Zaefferer, S.: Acta Mater. 156 (2018) 297.
85. An, D.; Krieger, W.; Zaefferer, S.: Int. J. Plast. 126 (2020) 102625.
86. An, D.; Zaefferer, S.: Int. J. Plast. 121 (2019) 244.
87. Kuzmina, M.; Herbig, M.; Ponge, D.; Sandlöbes, S.; Raabe, D.: Science 349 (2015) 1080.
88. Herbig, M.; Kuzmina, M.; Haase, C.; Marceau, R. K. W.; Gutierrez-Urrutia, I.; Haley, D.; Molodov, D. A.; Choi, P.; Raabe, D.: Acta Mater. 83 (2015) 37.
89. Zhou, X.; Kamachali, R. D.; Boyce, B. L.; Clark, B. G.; Raabe, D.; Thompson, G. B.: Acta Mater 215 (2021) 117054.
90. Darvishi Kamachali, R.; Kwiatkowski da Silva, A.; McEniry, E.; Ponge, D.; Gault, B.; Neugebauer, J.; Raabe, D.: npj Comput. Mater. 6 (2020) 1.
91. Gault, B.; Breen, A. J.; Chang, Y.; He, J.; Jägle, E. A.; Kontis, P.; Kürnsteiner, P.; Kwiatkowski da Silva, A.; Makineni, S. K.; Mouton, I.; Peng, Z.; Ponge, D.; Schwarz, T.; Stephenson, L.T.; Szczepaniak, A.; Zhao, H.; Raabe, D.: J. Mater. Res. 33 (2018) 4018.
92. Souza Filho, I. R.; Kwiatkowski da Silva, A.; Sandim, M. J. R.; Ponge, D.; Gault, B.; Sandim, H. R. Z.; Raabe, D.: Acta Mater. 166 (2019) 178.
93. Tarzimoghadam, Z.; Rohwerder, M.; Merzlikin, S. V.; Bashir, A.; Yedra, L.; Eswara, S.; Ponge, D.; Raabe, D.: Acta Mater. 109 (2016) 69.
94. Koyama, M.; Springer, H.; Merzlikin, S. V.; Tsuzaki, K.; Akiyama, E.; Raabe, D.: Int. J. Hydrogen Energy 39 (2014) 4634.
95. Sun, B.; Lu, W.; Gault, B.; Ding, R.; Makineni, S. K.; Wan, D.; Wu, C.-H. H.; Chen, H.; Ponge, D.; Raabe, D.: Nat. Mater. (2021) 1629.
96. Li, Z.; Pradeep, K. G.; Deng, Y.; Raabe, D.; Tasan, C. C.: Nature 534 (2016) 227.
97. Liu, C. C.; Li, Z.; Lu, W.; Bao, Y.; Xia, W.; Wu, X.; Zhao, H.; Gault, B.; Liu, C. C.; Herbig, M.; Fischer, A.; Dehm, G.; Wu, G.; Raabe, D.: Nat. Commun. 12 (2021) 5518.
98. Koyama, M.; Akiyama, E.; Lee, Y. K.; Raabe, D.; Tsuzaki, K.: Int. J. Hydrogen Energy 42 (2017) 12706.
99. Evertz, S.; Kirchlechner, I.; Soler, R.; Kirchlechner, C.; Kontis, P.; Bednarcik, J.; Gault, B.; Dehm, G.; Raabe, D.; Schneider, J. M.: Mater. Des. 186 (2020) 108327.
100. Nandy, S.; Tsai, S.-P. P.; Stephenson, L.; Raabe, D.; Zaefferer, S.: J. Magnes. Alloy. 9 (2021) 1521.
101. Palanisamy, D.; Raabe, D.; Gault, B.: Acta Mater. 174 (2019) 227.
102. Ma, D.; Grabowski, B.; Körmann, F.; Neugebauer, J.; Raabe, D.: Acta Mater. 100 (2015) 90.
103. Wu, X.; Li, Z.; Rao, Z.; Ikeda, Y.; Dutta, B.; Körmann, F.; Neugebauer, J.; Raabe, D.: Phys. Rev. Mater. 4 (2020) 33601.
104. Luo, H.; Li, Z.; Raabe, D.: Sci. Rep. 7 (2017) 1.
105. Luo, H.; Li, Z.; Lu, W.; Ponge, D.; Raabe, D.: Corros. Sci. 136 (2018) 403.
106. Luo, H.; Lu, W.; Fang, X.; Ponge, D.; Li, Z.; Raabe, D.: Mater. Today 21 (2018) 1003.
107. Korte-Kerzel, S.; Hickel, T.; Huber, L.; Raabe, D.; Sandlöbes-Haut, S.; Todorova, M.; Neugebauer, J.: Int. Mater. Rev. (2021) 1. https://doi.org/10.1080/09506608.2021.1930734
108. Sedighiani, K.; Shah, V.; Traka, K.; Diehl, M.; Roters, F.; Sietsma, J.; Raabe, D.: Int. J. Plast. 146 (2021) 103078.
109. Stoffers, A.; Barthel, J.; Liebscher, C. H.; Gault, B.; Cojocaru-Mirédin, O.; Scheu, C.; Raabe, D.: Microsc. Microanal. 23 (2017) 291.
110. He, J.; Zenk, C. H.; Zhou, X.; Neumeier, S.; Raabe, D.; Gault, B.; Makineni, S. K.: Acta Mater. 184 (2020) 86.
111. Makineni, S. K.; Kumar, A.; Lenz, M.; Kontis, P.; Meiners, T.; Zenk, C.; Zaefferer, S.; Eggeler, G.; Neumeier, S.; Spiecker, E.; Raabe, D.; Gault, B.: Acta Mater. 155 (2018) 362.
112. Yu, Y.; Zhang, S.; Mio, A. M.; Gault, B.; Sheskin, A.; Scheu, C.; Raabe, D.; Zu, F.; Wuttig, M.; Amouyal, Y.; Cojocaru-Mirédin, O.: ACS Appl. Mater. Interfaces 10 (2018) 3609.

113. Stephenson, L. T.; Szczepaniak, A.; Mouton, I.; Rusitzka, K. A. K.; Breen, A. J.; Tezins, U.; Sturm, A.; Vogel, D.; Chang, Y.; Kontis, P.; Rosenthal, A.; Shepard, J. D.; Maier, U.; Kelly, T. F.; Raabe, D.; Gault, B.: Cond.-Mat. Mtrl.-Sci. 1 (2018). http://arxiv.org/abs/1805.10836

114. Mianroodi, J. R.; Svendsen, B.: J. Mech. Phys. Solids 77 (2015) 109.

115. Mianroodi, J. R.; Shanthraj, P.; Svendsen, B.; Raabe, D.: Materials 14 (2021) 1787.

116. Breen, A. J.; Stephenson, L. T.; Sun, B.; Li, Y.; Kasian, O.; Raabe, D.; Herbig, M.; Gault, B.: Acta Mater. 188 (2020) 108.

117. Yan, F.; Mouton, I.; Stephenson, L. T.; Breen, A. J.; Chang, Y.; Ponge, D.; Raabe, D.; Gault, B.: Scr. Mater. 162 (2019) 321.

118. Sun, B.; Ma, Y.; Vanderesse, N.; Varanasi, R. S.; Song, W.; Bocher, P.; Ponge, D.; Raabe, D.: Acta Mater. 178 (2019) 10.

119. Sun, Z.; Tan, X.; Wang, C.; Descoins, M.; Mangelinck, D.; Tor, S. B.; Jägle, E. A.; Zaefferer, S.; Raabe, D.: Acta Mater. 204 (2021) 116505.

120. Stechmann, G.; Zaefferer, S.; Raabe, D.: Simul. Mater. Sci. Eng. 26 (2018) 045009.

121. Bastos, A.; Zaefferer, S.; Raabe, D.: J. Microsc. 230 (2008) 487.

122. Sedighiani, K.; Diehl, M.; Traka, K.; Roters, F.; Sietsma, J.; Raabe, D.: Int. J. Plast. 134 (2020) 102779.

123. Gallardo-Basile, F. J.; Naunheim, Y.; Roters, F.; Diehl, M.: Materials 14 (2021) 1.

124. Rao, Z.; Ponge, D.; Körmann, F.; Ikeda, Y.; Schneeweiss, O.; Friák, M.; Neugebauer, J.; Raabe, D.; Li, Z.: Intermetallics 111 (2019) 106520.

125. Lu, W.; Liebscher, C. H.; Dehm, G.; Raabe, D.; Li, Z.: Adv. Mater. 30 (2018) 1804727.

126. Lu, W.; Liebscher, C. H.; Yan, F.; Fang, X.; Li, L.; Li, J.; Guo, W.; Dehm, G.; Raabe, D.; Li, Z.: Acta Mater. 185 (2020) 218.

127. Wang, Z.; Lu, W.; Zhao, H.; Liebscher, C. H.; He, J.; Ponge, D.; Raabe, D.; Li, Z.: Sci. Adv. 6 (2020) eaba9543.

128. Moravcik, I.; Hadraba, H.; Li, L.; Dlouhy, I.; Raabe, D.; Li, Z.: Scr. Mater. 178 (2020) 391.

129. George, E. P.; Raabe, D.; Ritchie, R. O.: Nat. Rev. Mater. 4 (2019) 515.

130. Otto, F.; Dlouhý, A.; Pradeep, K. G. G.; Kuběnová, M.; Raabe, D.; George E. P. P.: Acta Mater. 112 (2016) 40.

131. Su, J.; Raabe, D.; Li, Z.: Acta Mater. 163 (2019) 40.

132. Wang, Z.; Lu, W.; Raabe, D.; Li, Z.: J. Alloys Compd. 781 (2019) 734.

133. Lu, W.; Herbig, M.; Liebscher, C. H.; Morsdorf, L.; Marceau, R. K. W.; Dehm, G.; Raabe, D.: Acta Mater. 158 (2018) 297.

134. Morsdorf, L.; Mayweg, D.; Li, Y.; Diederichs, A.; Raabe, D.; Herbig, M.: Mater. Sci. Eng. 771 (2020) 138659.

135. Herbig, M.: Scr. Mater. 148 (2018) 98.

136. Zachariah, Z.; Balachandran, S.; Liu, Z.; Pourzal, R.; McCarthy, S. M.; Hall, D. J.; Fischer, A.; Raabe, D.; Herbig, M.: J. Arthroplasty. 36 (2021) 2603.

137. Zhao, H.; Huber, L.; Lu, W.; Peter, N. J.; An, D.; De Geuser, F.; Dehm, G.; Ponge, D.; Neugebauer, J.; Gault, B.; Raabe, D.: Phys. Rev. Lett. 124 (2020) 106102.

138. Seol, J. B.; Bae, J. W.; Li, Z.; Chan Han, J.; Kim, J. G.; Raabe, D.; Kim, H. S.: Acta Mater. 151 (2018) 366.

139. Raabe, D.; Sandlöbes, S.; Millán, J.; Ponge, D.; Assadi, H.; Herbig, M.; Choi, P. P.: Acta Mater. 61 (2013) 6132.

Department of Structure and Nano-/Micromechanics of Materials (SN)

G. Dehm

Scientific mission and concepts

Mission

We perform basic research interlinking mechanical, and since 2019, the electrical properties of materials with their microstructure and constitution. Establishing such links requires multiscale approaches in materials characterization and property measurement. The gained information provides a strong basis for designing optimized material microstructures and systems with respect to improved lifetime in harsh environments, reduced energy consumption in transportation or higher efficiency in energy applications. While obtaining global properties is often well established, accessing properties that emerge at small dimensions from ca. 100 micrometres down to a few nanometres requires novel test strategies and protocols. We place our focus on adapting and developing reliable miniaturized experiments to obtain precise information on mechanical and electrical properties at these small length scales [1-7]. The high spatial resolution of our nanoindentation and 4-point probe based mechanical and electrical experiments, respectively, provide us access to the properties of individual phases in multiphase materials, which are often so far unexplored. Even more challenging are the complex interactions between different classes of imperfections such as between dislocation and grain boundaries [9-11] or dislocations and point defects [12-15], which is another focus of our work. As materials are always exposed to certain environmental conditions in applications, we have recently extended our small scale mechanical experimental portfolio to low (-150 °C) and elevated temperatures (800 °C), to variable strain rates ($10^{-4}\,s^{-1}$ - $10^{4}\,s^{-1}$) and also to hydrogen exposure – the latter in the new ERC funded group Hydrogen Mechanics and Interfaces (J. Duarte Correa). Combining our small-scale mechanical tools with advanced electron microscopy techniques and X-ray diffraction/synchrotron radiation methods allow us to control boundary conditions and to elucidate underlying deformation mechanisms. Similarly, *in situ* electrical characterization permits us to study site specific various families of defects down to a single coincidence site lattice grain boundary [16]. Understanding defects requires knowledge of their crystallography, chemical composition, bonding, and atomic structure. We use advanced scanning transmission electron microscopy (STEM) techniques, correlative electron microscopy and atom probe tomography (APT, often in collaboration with the MA department) to obtain this information [17-28]. Image simulations, machine learning, and atomistic simulations often in close collaboration with the CM department complemented by our new ERC funded group Atomistic Modelling of Material Interfaces (T. Brink) guide us with the necessary data interpretation [11, 13, 23, 26, 27, 29-31].

Our materials portfolio stretches from intermetallic materials [32-49] to chemically complex alloys / high entropy alloys (CCA/HEA) [12, 21, 50-56] and thin films [4, 25, 29, 52, 57-69] with specific aims. For example, next generation titanium aluminides for aero-engines target at extreme reductions in CO_2 and NO_x emission, while iron aluminides show great potential as an easy-to-recycle material for stationary engines and critical components in wind turbines. Our research often begins with establishing ternary phase diagrams, as literature lacks the requisite accurate data, critical for obtaining specific phases and microstructures. For CCA/HEA, we aim at improving high temperature strength by tailoring the microstructure towards those of superalloys and elucidate their resistance against hydrogen in the near future. An alternative approach to prevent hydrogen embrittlement of alloys is impeding hydrogen ingress by applying diffusion barrier coatings. This route we examine in a consortium with the independent research group Nanoanalytics and Interfaces (C. Scheu) at the MPIE. We test the impact of hydrogen on mechanical properties as well as its impact on nanostructure. In addition, we also elaborate on model materials, such as macroscopic bi-crystals with the help of the MPIE's central synthesis facility or elemental metal films fabricated in-house by SN department in a deposition cluster [10, 25, 59, 70, 71]. Those model materials are employed for very specific microstructure-property aspects, like the question if phase transformations of grain boundaries exist in pure elemental systems and how alloying additions would influence the transformation [19, 22, 23, 27, 28].

Department structure and laboratories

The department currently consists of 7 research groups closely interacting on our department's mission. The research groups Intermetallic Materials (M. Palm and F. Stein) and Thin Films & Nanostructured Materials (M. Ghidelli until Jan. 2020, J. Best since April 2020) are central to our and the institute's materials portfolio. They operate a modern thermodynamic laboratory, and thin film deposition facilities (with GO department), respectively. The 2013 established group Nano-/ Micromechanics of Materials (C. Kirchlechner until Dec. 2019, J. Best since April 2020) is complemented by the two new groups Hydrogen Mechanics and Interfaces (J. Duarte Correa, since 2019) and Nanomechanical Instrumentation and Extreme Nanomechanics (R. Ramachandramoorthy, since July 2020). This puts us now in a unique position for small-scale testing under hydrogen exposure with an in-house developed electro-chemical cell for back-side charging [7] (see p. 66), and developing new instrumentation for an expanded temperature and strain rates (≤10^4 s^{-1}) regime and variable gas environments (see p. 56). The group Advanced Transmission Electron Microscopy (C. Liebscher) hosts our aberration corrected TEM/STEM instruments (one probe corrected, one image corrected) as well as two conventional TEMs and specimen preparation facilities for conventional techniques and focussed ion beam (FIB) methods. One major scientific objective revolves around the ERC Advanced Grant GB-Correlate (G. Dehm) aiming at resolving and understanding grain boundary phase transitions in pure and alloyed metals. This task is now supported by the recently established group Atomistic Modelling of Material Interfaces (T. Brink, since 01/2020) financed by the ERC Advanced Grant, which also supports the group Hydrogen Mechanics and Interfaces (J. Duarte Correa, since 09/2019). This group performs atom probe tomography (APT) measurements on the chemical composition at grain boundaries and maps hydrogen in materials; the latter often in cooperation with the MA department. The activities of the former research group Nanotribology (S. Brinckmann until 12/2019) is now integrated in the group Nano-/ Micromechanics of Materials. S. Brinckmann, who accepted a tenured position at Research Centre Jülich, still co-supervises ongoing activities.

the AvH postdoc Y. Yao is now full professor at Xi'an (Northwestern Polytechnical University). Several postdocs became assistant or associate professor in China (W. Guo, W. Xia, W. Lu) and India (M. Kini, A. Kumar Saxena). We also have a large share of former postdocs and PhD students entering industry (e.g. steel industry, electronics industry, and companies for scientific instrumentation).

We support our department members' career development by personal interview trainings, offering external soft skill courses and a dedicated MPIE mentoring program (see p. 99) for selected female postdoc & staff scientists.

The high quality of our team members' research is also well documented by several awards. Examples for our PhD students are the selection for participation in the Lindau Nobel Laureate Meeting 2019 (N. Peter), the travel award of the Wilhelm und Else Heraeus Foundation (L. Frommeyer, 2020), and a Best Poster Award for S. Ahmet for her excellent work on grain boundaries in aluminium at the multinational microscopy conference MC2021. Z. Kahrobaee and M. Palm received the Editor´s Choice Award of the Journal of Phase Equilibria and Diffusion for their paper "Critical Assessment of the Al-Ti-Zr System" [42]. Last but not least the ERC Advanced Grant GB-CORRELATE (G. Dehm) (see p. 49), for which the sole selection criterion is excellence, was granted in 2018 and finances with 2.5 Mio€ for 5 years a large portion of the department's research work on grain boundary phase transitions.

Fig. 1: *The SN department and independent research group Nanoanalytics & Interfaces at the joint 2020 retreat in Lingen. Photo A. Frank.*

Personal development, careers and awards

Changes in our department's research groups are strongly linked to the excellent career opportunities of our scientists. C. Kirchlechner was appointed as tenured full professor and institute director at Karlsruhe Institute of Technology (KIT), S. Brinckmann became a permanent group leader at the Research Centre Jülich, and M. Ghidelli obtained a permanent staff scientist position at CNRS (Sorbonne, France). All three remain involved in co-supervision of PhD students who started under their guidance at SN department. Two former Alexander von Humboldt (AvH) awarded postdocs, S. Lee and X. Fang became group leaders at KIT and TU Darmstadt, while

Our international department members are often supported by prestigious fellowships from different countries and societies. In Germany, best known is the Alexander von Humboldt foundation, where excellent researchers are granted 1 to 2 year-long research stays in Germany. In 2020, D. Chatain, from CINAM – Centre Interdisciplinaire de Nanoscience de Marseille (France) received the Humboldt Research Award for established faculties and Y. Zhu from North Carolina State University (USA) the Friedrich Wilhelm Bessel Research Award for emerging faculties. Furthermore, Alexander von Humboldt Postdoc Fellowships were awarded to X. Fang, S. Lee, Y. Yao, X. Zhou (jointly with MA department), D. Xie, N. Cheng (jointly with Nanoanalytics & Interfaces), and Carvalho Silva.

Third party research projects

The SN department is actively integrated in several German Science Foundation (DFG) funded collaborative research centers, which align with the department's research mission. We are partners of: SPP 2006 Compositionally Complex Alloys - High Entropy Alloys (CCA - HEA), SPP 1594 Topological Engineering of Ultra-Strong Glasses (ended 2020), TRR188 Damage Controlled Forming Processes, and the SFB 1394 Structural and Chemical Atomic Complexity: From Defect Phase Diagrams to Material Properties (started 2020). Within the topic of HEA/CCA we also had until 12/2020 a joint French-German project on the stability of high entropy alloys (HEA) thin films with partners in Marseille (CNRS), Antipolis (U Mines Tech), and Bochum (RUB). Part of our hydrogen activities are funded in a joint project with the Leibniz Institute in Greifswald, Helmholtz Center HEREON in Geesthacht, and the independent research group of C. Scheu at the MPIE. Within the European Union's Horizon 2020 research and innovation programme the department is involved in the project ADVANCE, which is part of the Clean Sky 2 joint undertaking (see p. 88). Since 2021, we are part of the new Eurostars project – HINT. The ERC Advanced Grant GB-CORRELATE drives to a large extend our research on phase transformations of grain boundaries and their impact on material properties (see p. 49). Furthermore, we perform basic research projects with international scientific and European industrial partners. These projects are typically funded by the corresponding national state authorities such as the ministry of science or economics. Within the Max Planck Society we are connected to several institutes in the Max Planck PhD school Surmat and the Big Max activity on Big-Data-Driven Materials Science (see p. 82). The SN department also hosts 2 international Max Planck Partner groups led by excellent previous department members. The group *Designing Damage Tolerant Functional Oxide Nanostructures* (head: N. Jaya Balila, see p. 32) aims at improving the fracture resistance of brittle materials by incorporating interfaces. The external research group *Microstructure Design of High-performance Materials for Harsh Energy Applications* (head: J. Zavašnik, see p. 35) explores interactions of light elements (hydrogen, helium) with materials required for the hydrogen economy and fusion reactors, respectively.

Community service and teaching

The department supports the community by multiple reviewing and evaluation activities, serving in scientific and scholarly committees, participating as lecturers in conferences and schools, and by (co-)organizing workshops and conferences. The most prominent examples for conference (co-)organization are: "International Workshop on Advanced In Situ Microscopies of Functional Nanomaterials and Devices – IAMNano 2019" (G. Dehm, C. Liebscher, C. Scheu, B. Völker), "International Workshop on Laves Phases" (F. Stein 2019), "High entropy and compositionally complex alloys" (C. Liebscher, DPG 2019), Metals, Alloys and Intermetallics at the Microscopy Conference MC2019 (G. Dehm), "4th and 5th MSIT Winter School on Materials Chemistry" (M. Palm, F. Stein, 2020, 2021), "Nanobrücken 2020: A Nanomechanical Testing Conference" (G. Dehm, C. Kirchlechner, 2020), "TOFA 2020 – International Conference on Thermodynamics of Alloys" (F. Stein, M. Palm), "BiGmax Workshop 2020 on Big-Data-Driven Materials Science" (C. Liebscher with CM, and MA, 2020) and "BiGmax summer school „Harnessing big data in materials science from theory to experiment" (C. Liebscher with CM, 2021), "Intermetallics 2019 and 2021" (M. Palm, F. Stein), "International Conference on Metallurgical Coatings and Thin Films – ICMCTF" (J. Duarte, 2020, 2021), MRS Symposium "In Situ Mechanical Testing of Materials at Small Length Scales, Modeling and Data Analysis" (G. Dehm 2021).

Our close link to the nearby universities in Bochum (RUB) and Düsseldorf (HHU) is manifested in teaching courses on advanced TEM (G. Dehm, C. Liebscher), small-scale mechanical testing (G. Dehm supported by several group leaders and postdocs), and atomic resolved characterization (G. Dehm, C. Scheu), which can be also attended by our PhD students and postdocs. During the Corona pandemic, all lectures were given online. In addition, we make annual retreats with external guest scientists (except 2020 due to COV19), hold invited talks and discussion meetings with external scientists, and have a lively weekly joint SN and NG seminar series in addition to smaller group meetings. Since spring 2020, these seminars are all online.

The SN department is very well connected within the MPIE as seen by several multi-author papers where different techniques of the departments are connected, as for hydrogen related topics APT (MA), *ab initio* simulations (CM), Kelvin probe microscopy (GO), together with our TEM and small-scale testing capabilities.

The next chapter summarizes our main scientific breakthroughs followed by a more focused report on the scientific achievements of the research groups of the SN-department.

Main scientific breakthroughs

We have gained new insight in the existence of grain boundary (GB) phases in elemental metals and alloys by using thin films as model systems. Prominent examples are our Cu and Al films grown on (0001) α-Al_2O_3 substrates serving as ideal template systems due to their strong (111) texture with a large number of connected tilt grain boundaries ranging from low angle to high angle coincidence site lattice (CSL) GB. The strong (111) texture permits to resolve the atomic grain boundary structure by STEM/TEM with subatomic precision for a variety of GBs. Complementary molecular dynamic (MD) simulations are used to verify/predict stable grain boundary phases. We observed for the first time experimentally the coexistence of two different atomic motifs for Cu for a ∑19b grain boundary [23]. Very similar motifs exist also for the ∑37 grain boundary in Cu [27]. MD simulations confirm the observed grain boundary phases to possess different excess properties (volume, stress) and different stability ranges depending on temperature and stress. This shows that even in elemental metals grain boundary phase transitions occur more frequently than originally assumed! In order to tackle their influence on material properties, in 2019 we initiated high-resolution electrical measurements performed at room temperature. To obtain the resistivity of single grain boundaries we had to develop a novel approach with high position accuracy and so far unreached electrical sensitivity [1]. With this new tool we managed to detect a clear correlation between grain boundary excess volume and grain boundary resistivity for different CSL type grain boundaries in Cu [16]. Along the same lines, our micromechanical experiments on twin boundaries in Cu and Ag accompanied by MD simulations with the CM department reveal that a breakthrough stress for transmissible dislocations is in the range of shear stresses required for cross-slip [9-11, 64]. However, the maximum length of the mobile dislocation segment controls the required stress for dislocation motion rather than the “breakthrough stress” of the twin boundary [9, 10, 64]. Confining the dislocation by multiple finely spaced twins significantly contributes to the stress increase of nanotwinned materials [10].

Interaction of dislocations with lattice defects has been advanced by our studies on FeCoCr(Mn)Ni HEA and hydrogen charged Fe-Cr alloys. The jerky dislocation motion observed by *in situ* TEM straining experiments in the FeCoCr(Mn)Ni HEA [12, 13], does not require short range order. We could exclude short range order experimentally by advanced STEM, STEM-EDS and APT within the resolution limits [12]. MD simulations revealed that even with random atomic arrangements local fluctuations in the Peierls stress exert pinning points of different strength for dislocations [13]. In this collaboration within the SPP 2006 HEA/CEA with the TU Darmstadt we observed that strongest pinning points arise if Co atoms possess a Cr rich atomic neighbourhood within the random lattice occupation [13].

Hydrogen as a future energy source requires a fundamental understanding of hydrogen embrittlement processes. This we address by localized nanomechanical testing [7, 14, 15]. Currently single grains with different orientations in bcc Fe-Cr steels are in our primary focus. We found that the easier dislocation nucleation with diffusible hydrogen leads to an increase in dislocation density and thus a hardness increase compared to hydrogen-free Fe-Cr. The results are in accordance with the defectant theory predicting a reduction in dislocation line energy under hydrogen. In contrast to other studies in literature, we could prove that the Young’s modulus remains unaffected by the hydrogen ingress. This was only possible due to our novel back-side charging electrochemical cell developed in-house [7].

We made also progress in tuning the microstructure for improved high temperature materials required for CO_2 reduced transportation and energy generation. Novel superalloys based on the HEA system with the base elements AlFeNiTi where accomplished with creep resistance similar to commercial Ni-base superalloys [53, 54, 72]. For Ti-Al and Fe-Al based materials several highly accurate ternary phase diagrams have been developed in the last years [38, 42, 73-78]. While this is only a selection of most prominent examples, further details and examples are listed in the next sections under the individual research groups.

Scientific groups

Advanced Transmission Electron Microscopy (C. Liebscher)

Group mission

The mission of this group is to advance modern techniques in the transmission electron microscope (TEM) to unravel the micro- and nanostructural origins responsible for the functionalization of materials. We develop novel 4D-scanning TEM (STEM) techniques to explore grain and phase orientations in nanostructured alloys [30, 43], advance *in situ* techniques to observe the dynamic evolution of complex alloys under an applied strain [12, 79] or temperature [21, 80] and novel data analysis algorithms for improving atomic resolution imaging and automating its analysis [81, 82]. We apply the techniques to discover novel phenomena at grain boundaries down to atomic resolution [22, 23, 26, 28, 83], design new alloys with superior mechanical properties [20, 50, 53-55, 71, 84, 85] and push the boundaries in big data driven microscopy. Our goal is to understand the fundamental physical mechanisms active in materials and alloys at the atomistic level, building the foundation for the design of materials with superior functional [80, 86] and mechanical properties.

Grain boundary phase transformations

This is the biggest research topic within this group and covers many aspects across different material systems [22, 23, 26, 28, 59, 83]. The notion that grain boundaries, the interfaces separating individual crystals in a polycrystalline material, can exist in multiple phase states was established by thermodynamic concepts already more than 50 years ago. However, it was not possible to observe them at atomic resolution since grain boundaries are essentially confined, two-dimensional defects with a width of only a few atomic distances – buried in the material. Furthermore, they can adopt complex topographies [87] and only little is known about the discrete arrangement of atoms as well as solute species. We combine the synthesis of defined grain boundary geometries, site-specific sample preparation, atomic resolution characterization and atomistic modelling to discover the underlying nature of grain boundary phase transformations in metallic alloys.

It was so far only possible to explore grain boundary phase transformations in elemental metal systems by computer simulations. Together with T. Frolov (LLNL, USA), who is a world leading expert in atomistic modelling of grain boundary phases, we developed a direct correlation of atomic scale experimental observations and simulations of grain boundaries to explore novel interface phenomena. We could show experimentally for the first time that two grain boundary phases can occur at symmetric and asymmetric Σ19b(178)[11$\bar{1}$] tilt grain boundaries in an elemental Cu thin film [23]. As shown in the atomic resolution high angle annular dark-field (HAADF) STEM image of Fig. 2, the grain boundary structure transforms abruptly from the pearl phase (blue) to the domino phase (red). The same structures were found by grain boundary structure prediction based on an evolutionary algorithm and their difference in thermodynamic excess properties suggested that the two-phase pattern observed in experiment emerged from a first order transformation. By finite temperature MD simulations, we could establish that stresses are needed to stabilize the metastable domino phase. However, this could not explain the room temperature observations in experiment. The key to the answer of this question was the existence of a new, so far overlooked line defect, the grain boundary phase junction, which separates the two grain boundary phases (Fig. 2b). Temperature dependent MD simulations revealed that the migration of the phase junction is strongly temperature dependent and it is made responsible for kinetically trapping the transforming grain boundary phases for temperatures below ~500 K, which is ~200 K below the annealing temperature of the Cu thin film [23]. We are currently extending these observations to Σ37c [11$\bar{1}$] tilt grain boundaries in Cu, where we even witness a nanoscale dual-phase patterning of the grain boundary by similar grain boundary phases [27] (see section *Atomistic Modelling of Material Interfaces*, T. Brink). These observations require a paradigm-shift on how we treat grain boundaries and may open novel pathways to use the phase behaviour of interfaces to sculpt advantageous microstructures.

In related work, we have performed controlled segregation studies of similar Σ19b[11$\bar{1}$] tilt grain boundaries by doping them with Ta and Zr [22]. In the case of Ta, small nanosized Ta-particles are observed to form at the grain boundaries, which seem to pin them against movement during the annealing treatment. We could not observe any sign of Ta segregation to the grain boundary. This is different for Zr, where strong anomalous segregation is revealed. Our atomic resolution observations coupled to energy dispersive X-ray spectroscopy showed that Zr seems to disrupt the regular grain boundary structure in a Σ19b(178)[11$\bar{1}$] boundary, forming a glass-like grain boundary phase. A different behaviour was found for a faceted grain boundary, were Zr preferentially segregates into the facet junctions and disorders their core structure. These segregation-induced grain boundary transformations open a plethora of possibilities to further tune interface properties to tailor grain boundary mobility or the transport properties of interfaces.

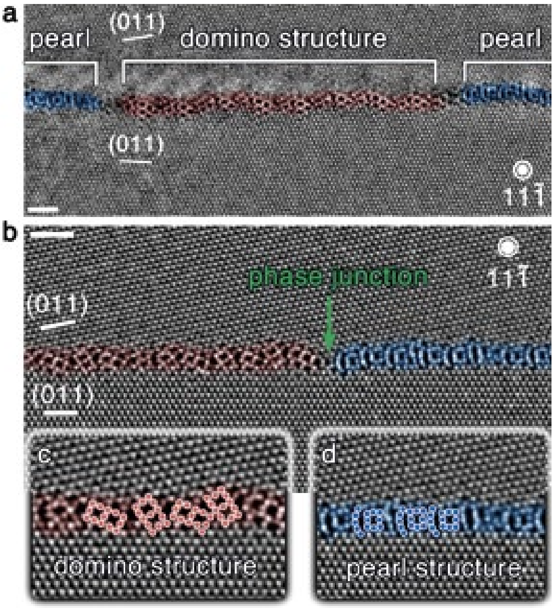


Fig. 2: *Atomic resolution HAADF-STEM images of two grain boundary phases pearl (blue) and domino (red) at an a) symmetric and b) asymmetric grain boundary in Cu. b) highlights the location of the grain boundary phase junction. c) and d) show details of the structural units of both grain boundary phases (from [23]).*

The segregation studies were extended to explore effects on asymmetric grain boundaries, which are rarely studied experimentally. We found that Ag segregation induces a nanofaceting transition at an asymmetric near Σ5(310) [001] tilt grain boundary in Cu [28, 83]. Atomic resolution STEM observations showed that the initial Ag-free grain boundary is asymmetric and adopts a rather complex structure. However, no facet formation was observed even after long term annealing at 800 °C for 120 h. The Ag-segregated boundary, which was annealed under similar conditions, showed distinct symmetric (210) facets that contained most of the Ag solutes. These facets, which were on the order of only several nm in size, adopted a kite-type structural unit, where the Ag atoms occupied the kite-tips and nearly filled an entire atomic column, as determined by STEM image simulations [28, 83]. The symmetric Ag-rich facets were separated by nm-sized Ag-lean asymmetric facets. In combination with MD simulations performed by T. Frolov (LLNL, USA), we could establish that Ag promotes the nucleation of the symmetric facets since the observed kite-structure is the lowest energy configuration. In recent work, we found that the facet evolution is strongly dependent on the Ag concentration by designing a diffusion couple geometry, where Ag diffusion was perpendicular to the tilt axis [28]. At low Ag concentration, the grain boundary remains flat and asymmetric and with increasing local Ag content, the emerging symmetric Ag-rich facets grow from several nm to tens of nm until a fully symmetric (210) segment emerges close to the Ag reservoir.

Compositionally complex alloys

The emergence of high entropy alloys (HEA), where five or more elements are mixed at near equal concentration, offers new opportunities for alloy design. We have employed our advanced characterization techniques to discover the fundamental deformation mechanisms in metastable, dual-phase $Fe_{50}Mn_{30}Co_{10}Cr_{10}$ (at.%) alloys with unmatched work hardening ability. Atomic resolution STEM imaging revealed that both the pre-existing face-centred cubic (fcc, γ) and hexagonal close packed (hcp, ε) domains in an as-processed alloy are composed of a nanolaminated structure, where hcp-nanolaminates form in fcc and vice versa [21, 79]. *In situ* straining in the STEM lead to the observation that the microstructure undergoes a dynamic forward transformation from $\gamma \rightarrow \varepsilon$ and backward from $\varepsilon \rightarrow \gamma$ upon straining, which we termed a bi-directional transformation induced plasticity effect. Ultimately, this leads to a hierarchical nanolaminated structure during deformation, which explains the excellent combination of strength and ductility of the alloys.

An extension of the outstanding room temperature mechanical properties was achieved in a similar alloy system of composition $Fe_{19.84}Mn_{19.84}Co_{19.84}Cr_{19.84}Ni_{19.84}C_{0.8}$ (at.%) by interstitial alloying with C [21]. We could show that nanotwinned regions in this alloy can be stabilized even to elevated temperatures of 700 °C by the formation of elongated $Cr_{23}C_6$-type nano-carbides. Atomic resolution STEM observations, STEM-EDS and *in situ* heating in the STEM reveal the underlying formation mechanisms of the nano-carbides. During annealing at 800 °C or 900 °C after cold rolling, the coherent twins in the nanotwinned regions undergo de-twinning forming nanosized incoherent twin boundary segments consisting of a dense dislocation network. Through thermal activation 9R phases are observed to nucleate at these incoherent segments, where the phase boundaries of the 9R phase ultimately serves as nucleation site of the nano-carbides. They in turn prevent further de-twinning and pin the twin boundaries from migrating, stabilizing the nanotwinned regions up to temperatures of 800 °C.

To advance the development of light weight, high temperature alloys, we have designed novel alloys in the composition space Al-Cr-Fe-Ni-Ti within the DFG priority program 2006 in collaboration with J. Schneider (RWTH Aachen University), U. Glatzel (University of Bayreuth) and the Intermetallics group (M. Palm, F. Stein). By combinatorial thin film screening we could efficiently identify suitable alloy candidates in the vast, unexplored composition space being comprised of a disordered bcc (A2) matrix and B2 and/or $L2_1$-type ordered precipitates [53, 54, 66]. Through compositional refinement and microstructural design we developed a low density alloy with composition $Al_{20}Cr_{20}Fe_{35}Ni_{20}Ti_5$ (at.%) and nominal density of 6.4 g/cm^3 with a yield strength of ~200 MPa at 900 °C and excellent creep properties at 800 °C [53].

The key to understand the mechanical properties in these highly concentrated solid solutions of HEAs is to observe

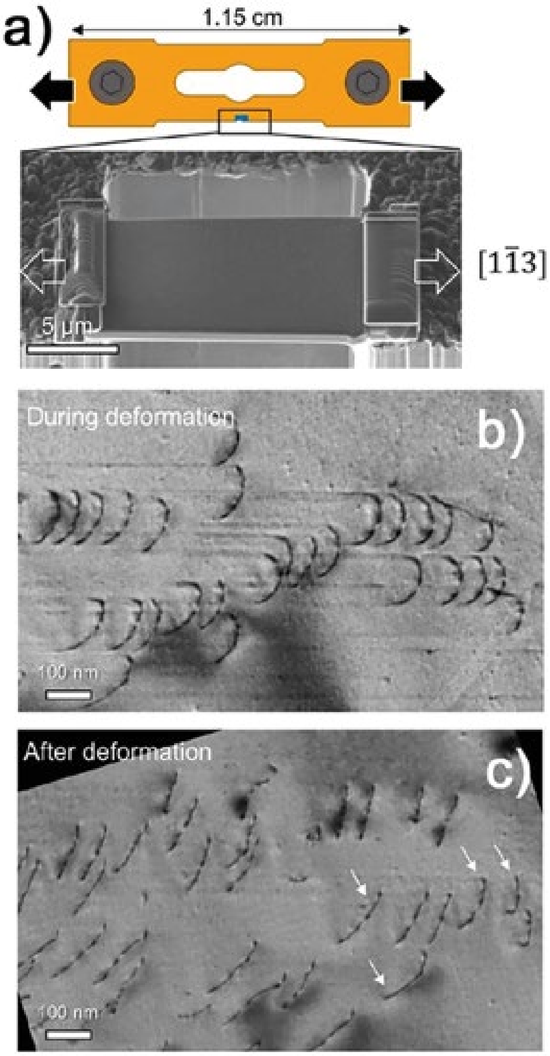


Fig. 3: *a) Custom built in situ straining inset and FIB prepared HEA sample prior to straining. b) Bowed dislocations during deformation. c) Partially bowed dislocations after straining indicated by arrows.*

how dislocations move through the lattice. We have developed a strategy to determine the size dependent micromechanical properties by *in situ* pillar compression in the scanning electron microscope (SEM) [88] and TEM [12] and correlated the mechanical properties to direct observations of dislocation glide by *in situ* straining in the TEM. We found a smaller size scaling exponent for a single crystal nominally equimolar FeMnCoCrNi alloy in comparison to other fcc metals reported in literature suggesting that solid solution hardening or a high lattice friction stress are dominant mechanisms. The direct observation of jerky dislocation motion and quantification of fluctuating local stresses acting on the dislocations (Fig. 3) lead to the conclusion that pinning sites are active. No traces of a local chemical short-range ordering was found by atomic scale STEM and APT characterization. An atomistic study performed in collaboration with the TU Darmstadt (K. Albe, D. Utt) revealed that local changes in the Peierls stress can already explain the jerky dislocation motion [13].

Big data and machine learning

With the advent of high-speed electron detectors new imaging modalities in the STEM emerged that enable to capture local crystallographic information with high spatial resolution. These 4D-STEM techniques, where in each probe position a full electron diffraction pattern is recorded, require also the development of new data treatment and analysis schemes due to the shear gargantuan amount of data produced, on the order of 10 GB to 200 GB per dataset. We have extended phase and grain orientation mapping by using a high-speed, high signal to noise CMOS-based electron detector. We could show that it does not only greatly improve the angular resolution, but also enables to locate nanotwins in a nanograined material [30] and opens the possibility to measure the local orientation of complex nano-phases embedded in a disordered matrix [43]. The analysis through commercial software packages of such large-scale datasets is often limited and we have therefore developed a Python-based open source, GPU-accelerated code with the aim to facilitate real time data analysis in the future. This will set the stage to combine *in situ* probing with 4D-STEM techniques to explore microstructure evolution dynamically with full crystallographic information.

Novel algorithms not only allow flexible and fast data analysis, but machine learning based approaches even provide the opportunity towards an autonomous data evaluation. Through collaborations within the BiGmax network with C. Freysoldt (CM department) and L. Ghiringhelli (FHI Berlin) we are developing supervised and unsupervised machine learning algorithms to segment and classify atomic resolution microscopy images. The unsupervised approach is based on local symmetry descriptors and is capable to automatically detect regions in images with deviating crystal structure or symmetry without prior knowledge of the underlying lattice structure [82]. This approach will be extended towards dynamic *in situ* observations and will be coupled to phase field models for being able to quantify and determine local properties of materials, here interface energies, while exposing the material to temperature in the electron microscope. The supervised deep learning approach is capable to classify image regions in atomic resolution STEM images and extracts local lattice constants and rotations. It is based on a convolutional neural network with simulated STEM images as ground truth training data. By coupling the classification to a Bayesian neural network, it is also possible to learn the location of defect and lattice disruptions, for example grain boundaries, through an uncertainty prediction. The supervised learning approach, which is so far based on Fourier space, will in a future collaboration with L Ghiringhelli be extended to a real-space classification, which is capable to also consider the intensity distribution of projected atomic columns in STEM images and hence local composition. The algorithm development is aimed to establish a self-learning and self-driving experimentation in the future.

Atomistic Modelling of Material Interfaces (T. Brink, since 01/2020)

Group mission: The group Atomistic Modelling of Material Interfaces was established in Jan. 2020 supported by the European Research Council (ERC GB-CORRELATE) to supplement the experimental work on grain boundaries in the department with atomistic computer simulations. Currently, we focus on investigating the thermodynamics of grain boundary phases using MD and density-functional theory (DFT) simulations. Several such grain boundary phases could be found using the HAADF-STEM imaging capabilities of the department, but the full three-dimensional structure as well as the thermodynamic excess properties of the grain boundaries remain inaccessible. Here, computer simulations can provide models of not only the phase stability, but also of e.g. mechanical behaviour of the grain boundary, which can affect macroscopic material properties such as grain growth or Hall-Petch hardening. Using this connection, the goal is to ultimately develop structure–property relations for grain boundaries.

Grain boundary phases

Most experimental evidence for GB phase transitions in metallic systems is indirectly inferred from changes in macroscopically measurable properties. While the fact that these changes can be measured underlines the importance of GB phase transitions, more direct evidence is required. Here, we worked on a $\Sigma 37c \langle 111 \rangle \{1\ 10\ 11\}$ tilt GB in copper in collaboration with the *Advanced Transmission Electron Microscopy* group as an example which exhibits interesting phase behaviour. In both experiments on thin films and in computer simulations, two GB phases were found, which possess similar atomic motifs as for the $\Sigma 19b[11\bar{1}]$ tilt GB (compare Figs. 2 and 4). Free energy calculations using the quasi-harmonic approximation show that these phases exhibit a congruent transition (i.e., no change of GB plane) at 450 K under ambient pressure. One novelty is that the transition of the $\Sigma 37c \langle 111 \rangle \{1\ 10\ 11\}$ requires neither diffusion nor externally applied stress [27].

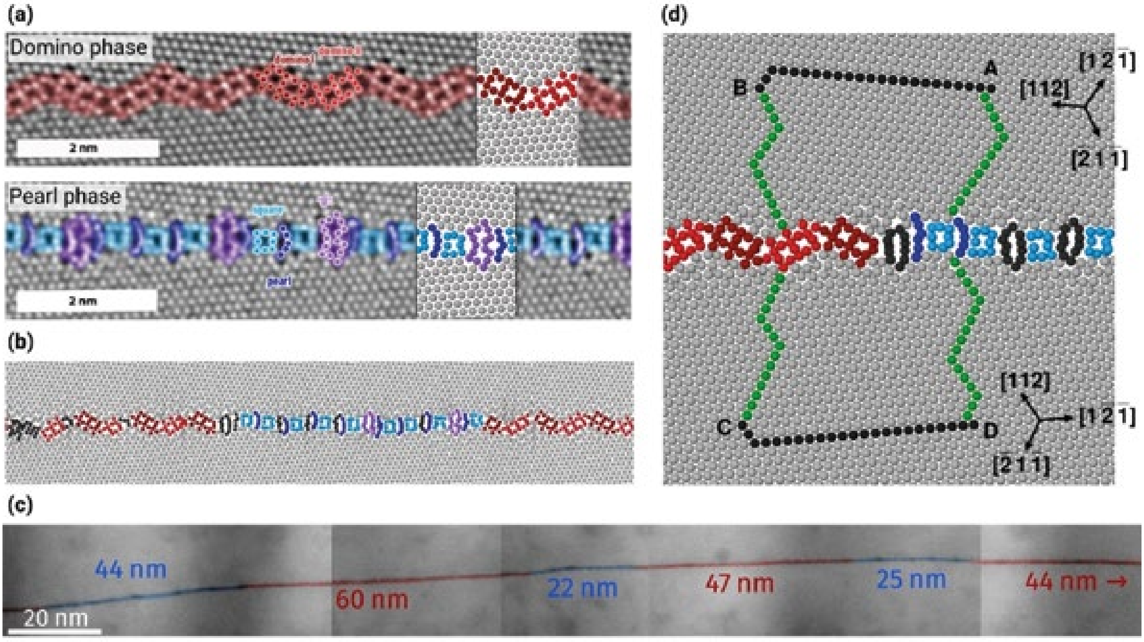


Fig. 4: *(a) Two grain boundary phases in a copper Σ37c ⟨111⟩ {1 10 11} tilt grain boundary found both experimentally and by computer simulation (insets). (b) Above 450 K a phase transition from Domino to Pearl phase occurs; here a simulation of the homogenous nucleation of the pearl phase is shown. (c) In experiment, the two phases form a pattern. (d) We analyzed the Burgers vector of the phase junction. From [27].*

Experimentally, though, a pattern of alternating phases is observed at room temperature (Fig. 4). Gibbs' phase rule excludes that such a microstructure can be stable and would lead to the assumption that the metastable phase remains in the system for kinetic reasons. Indeed, an analysis of the junction separating both grain boundary phases shows that this defect has a Burgers vector with a large screw component. Such line defects are expected to move comparatively slowly. Further analysis revealed that additional disconnections (dislocation-like defects in the GB), which exist to compensate asymmetry and/or a slight twist of the GB, can lead to repulsive interactions between defects and can potentially stabilize the phase pattern energetically. This shows that the complex interplay between defects and thermodynamics can lead to surprising effects in grain boundaries which need to be considered to fully understand these interfaces [27].

Presently, work is ongoing to broaden these analyses to other ⟨111⟩ tilt grain boundaries and fcc metals, such as aluminium. The goal is to differentiate general trends regarding the occurring phases and their stability from material-specific phenomena. In the near future, this work will also be extended from elemental metals to the addition of alloying elements. This is of particular importance for applications because temperature and stress state are almost always dictated by the use case, leaving only composition as a practical variable.

Structure–property relations at intergranular interfaces

The main practical importance of studying GB phases is the same as for bulk phases: The tailoring of material properties and the establishment of structure–property relations. We follow several different directions to elucidate these relationships.

It is well known that defects increase electrical resistivity, but the GB contribution is only known as an average value over larger sample volumes. First experiments on single GB resistivity measurements were performed in the *Thin Films & Nanostructured Materials* group (see p. 173) [1, 16]. While the resistivity could be measured as a function of crystalline misorientation and GB plane, the GB excess properties remain inaccessible to these experiments. We performed simulations on some of the high-angle boundaries studied experimentally and determined the stable phases at a high temperature approximately matching the annealing temperature of the experimental thin film. Using the derived GB excess properties, we could identify a correlation between excess volume or GB energy and resistivity [16]. We expect deeper, mechanistic insights from electronic structure calculations in collaboration with the CM department, which are currently ongoing.

Thermally activated grain growth is relevant for the evolution of a material's microstructure, while shear-coupled

GB motion can be a relevant mechanism of plasticity in fine-grained materials. Both are related to the GB mobility, which can be expressed as a tensor. In this project, we are interested in the influence of GB phase transitions on the mobility tensor. In many cases, grain boundaries migrate via the movement of disconnections, which can couple to an applied mechanical stress. Different GB phases can have different disconnection types, but even if these defects exhibit the same Burgers vector they can differ in nucleation energy and the energy barrier for their movement. Here, we study and characterize the defects responsible for the mobility and already found that the activation barriers of defects with the same Burgers vector can differ between different GB phases.

In the future, investigations on the influence of GB phase transitions, including for example faceting transitions, on dislocation nucleation and transmission are planned. Grain boundaries can act as obstacles to dislocations, thereby influencing macroscopic plasticity, or can act as sinks or sources for dislocations in fine-grained materials. We expect that this behaviour can be modified by GB phase selection.

Nano- and Micromechanics of Materials (C. Kirchlechner until 01/2020, J.P. Best since 04/2020)

Group mission

The overarching aim of the group is to quantitatively understand, predict and alter fundamental mechanisms governing plasticity, fatigue and fracture of materials. For this purpose, we isolate individual mechanisms – such as slip transfer of dislocations across a grain boundary – by performing small-scale mechanical experiments on samples under strict levels of geometrical and environmental control. But our work far surpasses the local measurement of strength and toughness: A central aspect of our research is to link the mechanical behaviour to the underlying and evolving defect structures (e.g. dislocation types and densities, local chemistry or phases, length and density of cracks and crack networks, etc.) which requires an advanced characterization toolbox with *in situ* capabilities.

An active and important mission of the group is method development. Over the last years 3D Laue microdiffraction techniques have been developed at the ESRF in Grenoble to determine decisive *in situ* material parameters (e.g. local strains, dislocation densities, grain boundary character, etc.) non-destructively with high spatial resolution (<1 μm). The 3D Laue micro diffraction activities at the SN department were led by C. Kirchlechner and will be continued at KIT. Additionally, *in situ* methodologies have been developed at the SN department for high-temperature measurement within an environmental chamber utilizing novel laser heating approaches, demonstrated for tensile testing of Cu thin films [3], along with further high-throughput tensile characterization of thin films [4]. The same environmental chamber used for this device is currently extended to microscale mechanical measurements under reactive environments (see p. 66), opening up exciting new challenges and possibilities in studying the interface between mechanics and chemistry during deformation and crack growth, for example. For these studies, new geometries are under active development to allow for stable crack growth, and will be exploited in the near future.

A developing theme within the group is the linking of local chemistry, imbued either through processing or environmental reaction in operando. In this space, environmental testing methodologies are being developed, including at temperatures ranging from −150 °C to 800 °C. For local chemistry variation, the deposition of thin films, which can be used to diffuse species to or along grain boundaries or dislocations is of active investigation.

Single and bi-crystal plasticity in metals

When single crystal sample sizes are taken down to the microscale, standard understandings of deformation no longer apply due to the probabilistic nature of dislocation plasticity. Therefore, motivated by sample size effects the group uses nanoindentation and micromechanical-based approaches to study the strength and deformation mechanisms of advanced material systems. This has been demonstrated for single crystalline Co-based Laves phases which have been studied as a function of crystal structure and composition [40, 41], but also for testing the critical resolved shear stress (CRSS) of basal and pyramidal I slip systems in pure Mg using a micro-shear testing geometry [6].

Within the framework of a recently-concluded SFB/Transregio TR188 project on dual-phase steels, significant progress has been made in understanding the CRSS of variant-containing martensite [89], and single crystal ferrite as a function of strain rate [90]. From these investigations, the activation volume for slip in ferrite was determined, along with insights into non-Schmid plasticity activation with increased strain rates. The CRSS for single phase regions of bainite have also been studied through an industrially funded collaboration. The gained information provides novel basic information and serves as inputs into crystal plasticity simulations with F. Roters in the MA department. In that work, phase identification of the complex bainitic steel microstructure was made through nanoindentation linked to machine learning clustering algorithms. As recent results have shown that indentation close to grain boundaries has a significant effect on the accuracy of such clustering approaches, we are currently trying to isolate boundary conditions where such machine learning approaches may be utilized.

Indeed, understanding deformation in the vicinity of grain and phase boundaries constitutes a significant body of work within the group, as important open questions currently exist on how GB structure and chemistry influences transmission mechanisms, and the local strength. Within this realm, the group has studied in detail Cu micropillars which contain a single GB. Coherent Σ3 (111) twin boundaries in Cu have been shown through extensive study to allow cross-slip-like transmission, leading to low transmission stresses and similar deformation behaviour to that of single crystals. However, when thin-films of Ag containing closely spaced nanotwin arrays were produced, micropillar compression experiments showed high strengthening due to dislocation bowing in each single crystal zone during the continuous slip [10, 64] (Fig. 5). Cross-slip in Cu is currently being extended to higher-energy incoherent Σ3 (211) twin boundaries, where the transmission stresses and mechanisms are being investigated for magnetron sputtered thin-film systems where such non-equilibrium boundaries can be produced in a controlled manner. Twin boundaries can also act as dislocation sources, while recently published work on Cu showed that spherical nanoindentation on such boundaries allows a source activation stress smaller than that needed for bulk single crystals, irrespective of indenter size, dislocation density and stacking fault energy [91, 92]. The role of chemistry on dislocation activation at, or transmission through, grain boundaries is a current focus using micropillar compression and high-throughput nano-scratching supported by advanced microscopy.

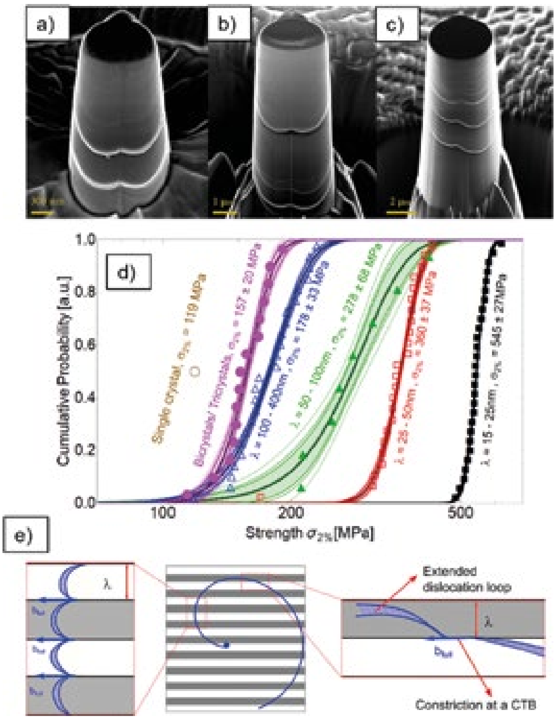


Fig. 5: *Dislocation plasticity at twin boundaries. Post mortem SEM micrographs of bi-crystal micropillars with nominal diameters of 1 μm (a), 3 μm (b), and 5 μm (c) compressed to a strain of 5-10 %. For Ag thin-films containing nanotwins, cumulative probability distributions of $\sigma_{2\,\%}$ strength data divided into twin spacing λ bins are shown (d). Proposed plasticity mechanism in closely-spaced nanotwin containing Ag thin films (e); dislocation loops close to a conjugated slip system are projected onto a pillar slip plane showing several constrictions at CTBs [10].*

Microscale fracture mechanics

Failure through fracture of engineering components and structures has an enormous cost to society. Such processes are initiated at small scales at atomically-sharp crack tips, where plasticity and decohesion processes are involved at the stress concentrations which develop under loading the notched component. Therefore, there is a significant link between local plasticity and fracture. Linear elastic fracture mechanics (LEFM), or fracture prior to plastic deformation, is an established method at small-scales in the group [93] and is used to study advanced materials and material microstructures such as grain boundaries [17].

For brittle materials, plasticity is limited due to the difficulty of activating dislocations in stiffly bound covalent systems, and therefore fracture occurs during the elastic region governed by LEFM. This has been demonstrated for a number of thin-film ceramic systems such as multi-metal carbides [67, 94], and AlN-based coatings [95]. Using these principles, the group has assessed the reliability of annealed metallic interconnect thin-films in an industrially funded basic research project, where the fracture toughness from single cantilever bending experiments is linked to the structure and chemistry of the initially amorphous Ni-P alloy as a function of temperature. Current work looks at linking the brittle-to-ductile transition as a function of temperature and strain rate for bulk-scale intermetallic systems, as for our project within the DFG-funded SFB 1394 focused on Laves phases in the Mg-Al-Ca system (see p. 76).

When fracture necessitates significant plastic deformation around the crack tip, then elastic plastic fracture mechanics (EPFM) is utilized. EPFM is at the frontier of understanding for micro-geometries, but has huge promise for understanding fundamental processes during fracture for tough materials systems. Within TR188, together with our advanced understanding of the CRSS in single crystal ferrite regions, novel cantilever-based micro-geometries have been developed to allow for EPFM analysis of microscale martensitic islands [96]. Similar approaches are currently used to perform local fracture of single grain boundaries in tungsten. Recent projects have also looked at white etching layers in steel rails [97, 98], together with an overarching aim of further developing EPFM based on optimization of the notch and testing geometry for improved reliability of EPFM tests [5].

Advanced testing of functional oxides

Oxides find broad applications as catalysts or in electronic components, however are generally brittle as dislocations are difficult to activate in the rigid covalent lattice. Here, the link between plasticity and fracture is critical for wide-scale application of functional oxide materials. Together with our Max Planck partner group *Damage Tolerant Functional Oxide Nanostructures* (head: N. Jaya Balila) at the IIT Mumbai (see p. 32) we have analyzed room temperature deformation in $BaTiO_3$

where near-theoretical strength is achieved for sub-micron pillars, and significant plasticity reported as the length scale increases past 1 µm [99]. Ongoing research looks at the temperature-dependent microscale fracture toughness of $BaTiO_3$ around the Curie temperature where a tetragonal to cubic phase change is expected. Additionally, we study sub-micron single crystal pillars of the transparent semiconductor Ga_2O_3 under variable strain rate conditions, to elucidate subtle transitions in active dislocation mechanisms during compression, which may be later linked to improved processability of the single crystal with applications in optoelectronic devices [100].

We also focus on how defect structures in oxides affect the functional properties. Together with collaboration partners in Darmstadt, we have determined that the dislocation structure formed under low-load indents into TiO_2 [101] can have a strong effect on the electrical resistivity. High temperature measurements are also ongoing on single crystals of various iron oxide phases. $Fe_{1-x}O$ (Wüstite) is a critical phase for a number of applications. In the CO_2-free H_2-based iron ore reduction, for example, $Fe_{1-x}O$ is the rate-determining intermediate phase in the production of iron for the steel industry. While for H_2 production by water-splitting, $Fe_{1-x}O$ is a potential photochemical material. For both of these processes, the transport mechanisms of Fe ions within $Fe_{1-x}O$ and therefore the atomic structure of $Fe_{1-x}O$ are key to understand. In studying how the variations in oxide stoichiometry affect the defect structure and mechanical properties of the Wüstite phase, mechanical investigations on bulk-scale single crystals are also here complimented by reactive magnetron deposited iron oxide thin films for precise control over both the structure and chemistry of the resultant phase.

Nanomechanical Instrumentation and Extreme Nanomechanics (XNano) (R. Ramachandramoorthy, since 08/2020)

Group mission

Pushing the envelopes of micro/nanomechanical testing to relevant, but so far unexplored strain rate and temperature combinations is one central mission of this new group. In addition, additive micromanufacturing across meso-, micro- and nanoscales to obtain novel structural and functional lattice structures constitutes our second goal. For an overview of the group's research focus and the motivating factors, refer also to the section on new research groups (p. 46). Given the unique challenges presented by small scale sample fabrication and their characterization under extreme loading conditions, a significant effort in our group is focused on instrumentation, methods and protocol development. It is our vision to use such novel infrastructure and testing methods to uncover the fundamental microstructure-property relationships of micro- and nanoscale materials under application relevant strain rates and temperatures, which currently remain largely unexplored.

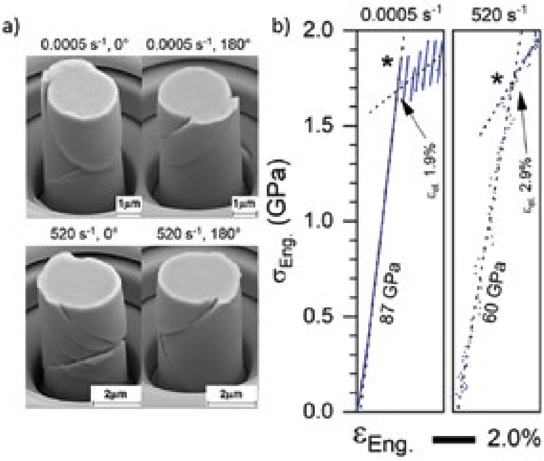


Fig. 6: *a) SEM images of bulk-metallic glass micropillars compressed at strain rates of 0.0005/s and 520/s and their corresponding stress-strain signatures are shown in b) [8].*

Dynamic micro- and nanomechanical testing

A fundamental understanding of the microscale deformation mechanisms during high-speed impacts is vital for developing protective technologies for demanding applications such as micrometeorite impact with spacecrafts, cold spray coating technology and microparticle impacts on aircraft turbines. Regardless, owing to the lack of testing platforms, the majority of micro- and nanomechanical testing focuses only on quasi-static testing [102]. To conduct micromechanical testing under extreme strain rates >10.000 s^{-1} with the simultaneous capture of high-speed load and displacement signatures (see Fig. 6 for bulk-metallic glass example [8]), we have performed extensive hardware modifications to an *in situ* micromechanical testing system [8, 103]. This includes the introduction of a high-speed voltage supply for activating a piezo-based actuator at high speeds (up to 100 mm/s), piezo-based load-sensing system and support electrical systems for high-rate signal generation (~25 MHz) and acquisition (~5 GHz). Additionally, protocols and methods specific for such extreme micromechanical testing are currently developed to address problems such as system resonances, inertia, wave propagation and low signal-to-noise ratio.

Nanomechanical testing is an invaluable tool for understanding the fundamental deformation processes in 1D and 2D materials such as nanowires and nanoparticles. But such nanoscale mechanical metrology even for quasi-static tests requires specialized testing platforms that are capable of applying displacements with nanometer resolution and resolving loads with nano-Newton sensitivity [104]. To address such stringent requirements, in conjunction with company partners and supported by EU funding, we are currently developing an *in situ* microelectromechanical system (MEMS)-based high-speed testing platform with electronics-based capacitive load sensing

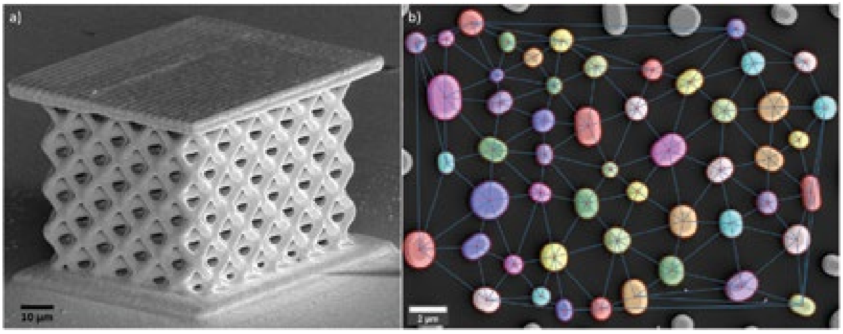


Fig. 7: *a) Full-metal copper micro-lattice fabricated using additive micro-manufacturing. b) Dewetted nickel microparticles post-processed for size, shape and density analysis.*

capability (~500 kHz bandwidth) and piezotube-based high speed actuation (up to 100 mm/s), to be used inside an SEM and TEM.

Additive micro-manufacturing

Full-metal micro/nano architectures have profound applications in metal MEMS based devices, MEMS packaging for energy absorption/impact resistance, photonics, plasmonics, catalysis and corrosion studies. Conventional additive manufacturing, though it guarantees geometric freedom and spatial control, is limited to a minimum feature size of 20 µm. Thus, there is a critical need to identify a method for 3D printing metals at the micro- and submicron scale (down to ~200 nm feature size) to create complex – and otherwise hard to reach – geometries (~1 µm to 1 mm overall dimensions) such as meso-/micro-/nano-lattices, microsprings etc. [105]. To address this requirement, we explore a localized electrodeposition-based additive micro-manufacturing technique (Fig 7a). In this method, an electrochemical ink with metal ions is pushed through a hollow microchanneled atom force microscopy (AFM) probe, which gets reduced onto a conductive substrate layer (working electrode). The voxel-by-voxel manner of metal deposition allows the fabrication of complex 3D microarchitectures with a voxel diameter of 200 nm to 15 µm, depending on the aperture size in the AFM probe. Using this method, any electrodepositable metal can be printed into arbitrary-shaped small-scale architectures and so far, we have successfully fabricated full-metal complex 3D copper micropillars and microlattices (see p. 64 for additional application of fluid-filled architecture). We will further investigate the suitability of such full-metal architectures towards energy absorption and mechanical band-gap engineering applications, using *in situ* and *ex situ* dynamic testing inside an SEM and under an optical microscope (see p. 66 for environmental chamber being developed for this purpose) respectively.

Dynamic thermomechanical characterization of dewetted microparticles

Another promising technique to obtain a large number of pristine micron scale metallic samples is using solid-state dewetting of thin films [63]. In collaboration with the *Thin Films & Nanostructured Materials* group in the SN department, we have developed specific protocols for dewetting nickel thin films to produce single-crystal nickel microparticles. To optimize the dewetting protocols we use python-based algorithms to identify the particle size, shape and density in an automated manner from SEM images illustrated in Fig. 7b. We plan to apply similar thin film dewetting methodologies to soon obtain iron and iron oxide microparticles. Currently, a high throughput mechanical testing study is underway to obtain the dynamic properties of such dewetted nickel particles at a variety of strain rates (10^{-4} s^{-1} to 10^{+4} s^{-1}) and temperatures (-150 °C to 800 °C) using the custom modified *in situ* micromechanical tester inside an SEM. We hope to uncover the deformation signatures of single crystal nickel under these extreme loading conditions using a combination of the thermal activation parameters extracted from the stress-strain signatures and post-test microstructural characterization using TEM and Kikuchi diffraction techniques.

Hydrogen Mechanics and Interfaces (M.J. Duarte Correa, since 09/2019)

Group mission

We correlate the microstructural properties of metals and alloys and their interaction with environmental factors, particularly hydrogen. A key element is the study of interfaces, since they are largely responsible for material strength. The final goal of our research is to minimize the deleterious effects of hydrogen and provide guidelines for hydrogen tolerant materials. The group was established in September 2019 supported by the European Research Council (ERC GB-CORRELATE).

The mechanical behaviour of a material can be significantly altered by the presence of hydrogen (Fig. 8), causing catastrophic and unpredictable failure, known as hydrogen embrittlement. This represents a significant challenge for the implementation of hydrogen as a sustainable energy source. The large infrastructure required for a hydrogen economy, as well as the use of structural materials in environments that promote hydrogen uptake, require the understanding of the complex embrittlement phenomena at the nano- and microscale level; failure often starts at this length scale with accumulated local hydrogen which can evolve into macroscopic damage. Our work is focused on the understanding of individual damage mechanisms by performing small-scale mechanical experiments. We target specific underlying and evolving microstructural features (Fig. 8) and their mechanical response upon hydrogen ingress. Mechanical characterization is performed at length-scales below a few **µm** by nanoindentation and related techniques, such as nanoscratching, pillar compression and fracture testing [7, 15]. However, as the analyzed volume becomes smaller, the risk of hydrogen desorption becomes more relevant. A particular challenge is then to provide a continuous hydrogen supply during mechanical loading to minimize desorption and the associated formation of strong hydrogen concentration gradients at the testing regions.

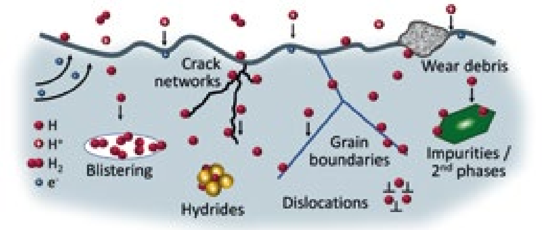


Fig. 8: *Schematic of hydrogen interactions with different microstructural features in metals.*

Instrumentation

We develop unique instrumentation and advance current tools to promote different atmospheres during mechanical testing and to introduce hydrogen into the material. We built two custom electrochemical cells for *in situ* hydrogen charging during nanoindentation, designated as "front-side" and "back-side" charging [7] mounted in a standalone nanoindenter. The front-side charging with the sample and indenter tip immersed into the electrolyte utilized similar concepts to existing setups. Due to direct contact of the tested surface with the electrolyte, hydrogen charging in the region of interest is fast, but has the disadvantage of often generating surface degradation. In the back-side charging approach, newly developed in the group, the analyzed surface is never in contact with the solution and the observed effects are only due to hydrogen. Hydrogen diffusion from the charged back-side towards the testing surface is quantified by permeation tests. Moreover, this unique method allows differentiating between the effects of trapped and mobile hydrogen, and performing well controlled measurements where different hydrogen levels can be monitored over time to consider hydrogen absorption, diffusion and release through the metal. We are currently implementing the back-side setup into a new instrumented nanoindentation platform inside a custom-made environmental chamber providing different atmospheres such as oxygen, inert or forming gases and dry air. In further developments, we aim to introduce this setup into a SEM.

Plastic deformation during hydrogen charging

A key question of this work is how hydrogen interacts with dislocations. The hydrogen influence on the plastic and elastic behaviour is studied at specific charging conditions using the back-side setup. Through nanoindentation we analyse the elastic modulus, hardness, incipient plasticity and dislocation nucleation, which when combined with pillar compression we further investigate the effect of hydrogen on the yield strength, i.e. insights into dislocation propagation and interactions. Current work is performed in model alloys to reduce the complexity of e.g. steels. We focus currently on Fe-Cr, Fe-Ni and Ni based alloys representative of body centred and face centred cubic structures. As an example, enhanced dislocation nucleation and hardness increase was observed while increasing the hydrogen uptake in Fe-Cr (Fig. 9 top). We established a linear relationship between the hardness increase and the hydrogen supply, and identified a hydrogen saturation level in Fe-Cr alloys. This hardening effect was modelled and attributed to an enhanced dislocation multiplication, pinning on the dislocation motion and an increase in lattice friction related to hydrogen.

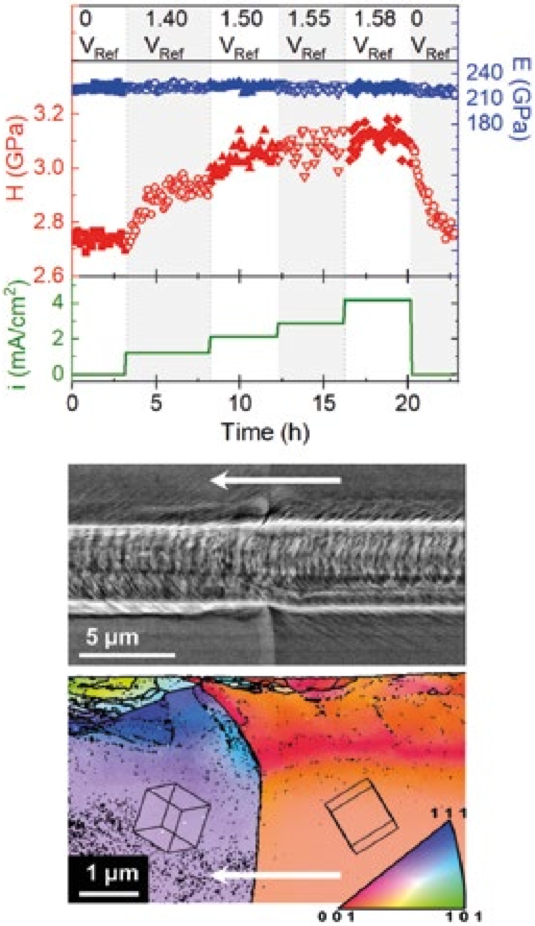


Fig. 9: *(Top) Hardness variation with current density (i.e. introduced hydrogen) and, (bottom) nano-scratch across a grain boundary and corresponding transversal inverse pole figure that shows recrystallization and the deformation extent. (All during cathodic polarization for hydrogen charging of a Fe-20 wt.% Cr alloy).*

Decohesion and failure at interfaces due to hydrogen

One of the most critical damage nucleation sites are interfaces. Most engineering and structural alloys contain a large density of grain and phase boundaries, affecting dislocation motion, generation and annihilation, and largely contributing to their mechanical strength. These interfaces can be seriously embrittled by hydrogen resulting in a significant degradation of the global mechanical properties. This prompts to question how the interface character, elemental segregation and hydrogen trapping behaviour influence failure and interface decohesion. We perform targeted measurements in model alloys and steels to probe this question. The important role of dislocation-interface interactions is initially examined by high-throughput scratch tests. The initiation of an intergranular crack is favoured if dislocation slip transmission is suppressed by the local stress incompatibility at the interface and enhanced by the accumulation of hydrogen. This might result in dislocation pile-ups at the interface and can be evidenced by a curved grain boundary after scratch testing (Fig. 9 bottom). Interface failure by crack initiation and propagation are further examined in specific decohesion experiments.

Hydrogen barrier coatings

In collaboration with the group Nanoanalytics and Interfaces (C. Scheu, MPIE), the Leibniz Institute in Greifswald, and the Helmholtz Center HEREON in Geesthacht, we study how to prevent or limit the hydrogen entry into structural alloys by the use of barrier coatings. Our initial studies reveal that bilayer Al-alumina coatings sustain exposure to 300 bar H_2 at 400 °C for 6 days; equivalent to Ar exposure under the same testing conditions. However, dewetting of the Al film occurs which indicates that pure alumina coatings are better suited. This is currently under investigation.

The mechanical findings in each project are complemented with other characterization techniques through multidisciplinary collaborations with all the departments at the MPIE and the group network. This includes especially thermal desorption spectroscopy and Kelvin probe measurements (M. Rohwerder, GO department) as well as APT studies (B. Gault, MA department).

Nanotribology (S. Brinckmann, until 12/2019)

Group mission

The investigation of microstructure evolution during wear of surfaces using well-defined and inert micrometre asperities that mimic the single asperities of macroscale components is the central mission of the *Nanotribology group*. These fundamental investigations of dry materials tribology address the irreversible microstructure mechanisms such as dislocation plasticity and twinning, grain refinement, as well as phase formation and dissolution [106-110].

Microstructure evolution during the initial stages of tribology

Understanding the transition from indentation to ploughing and the corresponding evolution of plasticity in austenite stainless steel for different grain orientations is one of our central research topics (Fig. 10).
We find that slip-step and pile-up evolution during the indentation segment influence plasticity during the subsequent ploughing segment [106]. Four stages can be seen during ploughing: first deformation by indentation, a transition region, steady-state ploughing at a rather constant depth, and finally the unloading segment. The lateral motion of the ploughing results in a break of deformation symmetry as documented by the pile-up and glide steps at the surface (Fig. 10). The deformation pattern is very distinct to that after indentation.

These findings highlighted the need to thoroughly understand the first contact of an asperity with the metal surface, and we hence extended the knowledge of nanoindentation and the microstructure evolution during this deformation stage. Towards this goal, we deformed grains with specific low indexed crystal orientation to

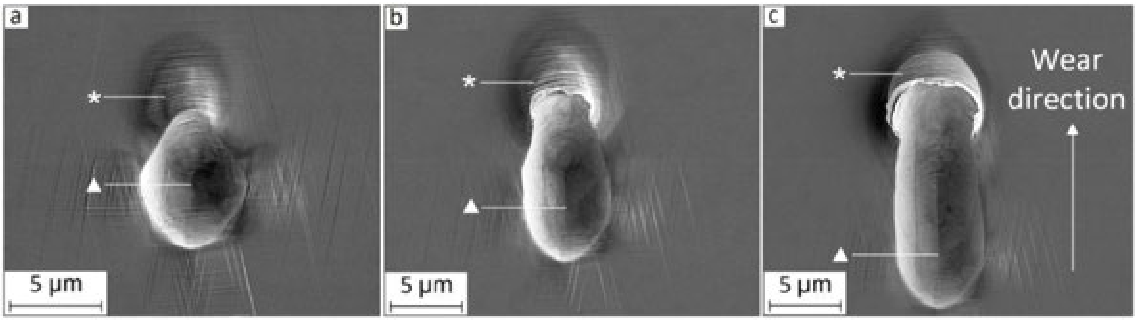


Fig. 10: *Secondary electron images of wear tracks with a 40 mN normal force in a [001] γ-Fe grain; a) 2 µm, b) 6 µm, c) 10 µm wear length. The white arrow in (c) denotes the wear direction for all tracks, the white asterisks and triangles mark the pile-up and the position of the initial contact, respectively.*

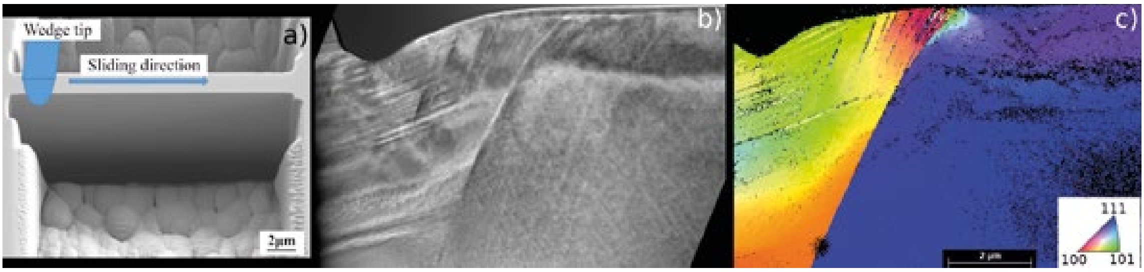


Fig. 11: *Tribology experiments on microwall: a) SEM image of microwall consisting of two twinned crystal orientations before deformation; b) dark-field TEM image of deformation and twin boundary and dislocation structure; c) Transmission Kikuchi diffraction (TKD) orientation map of the twin boundary and deformation in the left-hand grain.*

better understand the dislocation mediated plasticity through slip step analysis on different slip-plane inclinations [107]. We found that slip steps emerge first on slip planes starting from below the indenter towards the surface (positively inclined slip planes), while at a later deformation stage slip steps emerge from outside the indented zone towards the indent (negatively inclined slip planes) due to the change in surface topography. We calculated the resolved shear stress in the presence and absence of pile-ups and confirmed that the surface topography is the origin of the activation of the different set of glide planes [107]. We concluded that accounting for the surface topography evolution in experiments and simulations is essential in predicting the plastic slip activation during nanoindentation.

As the surface topography plays such an important role, a better understanding of the pile-up formation is warranted. We revealed by experiments that the shape of the pile-up changes at a critical inclination angle 55°-58° from radial plastic flow to circumferential plastic flow for the different grain orientations [108]. As the plastic flow of other activated slip-planes change similarly, the interaction of these slip-planes determines the final pile-up shape. These experimental findings, which are based on SEM surface topography and SEM-ECCI (electron channelling contrast imaging) analyses were augmented by finite element method (FEM) simulations to better understand the stress state during indentation and the influence of the topography. The FEM simulations reveal that the resolved shear stress inverts around this critical angle for the different grain orientations studied.

As the deformation of the microasperity transitions from indentation to wear, i.e. transitions from stationary to sliding contact, the conservation of contact area, elastic recovery, and the front pile-up development dominate the deformation mechanisms [109]. By investigating this transition in copper, cementite and austenitic steel, we concluded that the elastic recovery leads to an additional contact area at the backside of the asperity. The influence of the crystallographic orientation was found to be negligible during the initial increase in wear depth but significant during the later stages. Moreover, a partial elastic wear depth recovery was observed in ductile materials.

Evolution of twinning during wear

Twinning and the resultant microstructure evolution can lead to the formation of a hard surface layer that protects the material during continued tribological loading. We investigated the influence of wear direction on the deformation mechanisms in single austenite steel grains (Thermax) with {001} and {111} normal orientation and observe multiple intersecting twin structures below the wear track. The extent of deformation and the twin density was found to be dependent on the wear direction, normal load and asperity geometry. The competition of twinning and dislocation plasticity is the origin for the differences between grain orientations: some grains show predominantly twinning while others preferentially exhibit dislocation plasticity.

Simplifying the stress state during plasticity to improve the understanding of wear mechanisms

Single nano-/ microasperity sliding wear tests allow to separate the microcontacts and to study the origins of plastic evolution during the initial stages of tribology. We pre-remove material before scratching to obtain microwalls of finite width (approximating a 2-dimensional structure) (Fig. 11). This simplifies the stress state and allows to directly inspect the contact zone from the top and the side. After sliding on the microwalls, a misorientation jump forms underneath the wear track by formation of a grain boundary. The crystallographic misorientation reached 30° in some configurations, resulting in the formation of large angle grain boundaries. We unravelled the grain formation mechanism via slip-step and dislocation analysis by addressing dislocation arrangements at the grain boundary.

Thin Films and Nanostructured Materials (M. Ghidelli from 01/2019 until 01/2020, J.P. Best since 04/2020)

Group mission

We focus on the development and synthesis of novel nanostructured thin films, while exploring their physical and mechanical properties [1, 16, 52, 95]. Motivating the group's work is the requirement of novel high-performance thin films with superior structural and functional properties for advanced applications such as micro-/nanoelectronics, energy production, sensors and wear protection [25, 52, 57, 59, 60, 63, 95]. In particular, intrinsic but mutually exclusive structural properties such as high strength and ductility must be combined, but also the resistance to harsh conditions such as corrosive environments, wear, and high temperature be improved. Our research also includes thermal stability against decomposition, grain growth and dewetting.

Key for property control is the film architecture and microstructure. To afford control over the material properties the microscale porosity, atomic composition, average grain size, phase distribution, and layer/film thickness must be optimized. In achieving such a high level of control, the group leverages a unique physical vapor deposition cluster to perform magnetron sputtering and electron-beam deposition of both metals and ceramics. We further ongoing links on alloy development and novel alloy combinations forged together with the MA department, where novel high entropy alloys and amorphous metal thin films are deposited for high-strength high-toughness combination materials.

tensile tests on flexible substrates, and X-Ray diffraction as a function of temperature. From these experiments we have found that the content of Cu (at.%) influences the atomic arrangement, and the resulting mechanical properties and thermal stability, which we currently aim to link to the characteristic atomic-scale order through high-energy synchrotron measurements.

Moreover, we managed to fabricate complex film architectures from the $Zr_{100-x}Cu_x$ system, such as multilayers and amorphous-nanocrystalline composites, to investigate the role of interfaces and secondary phases as obstacles to shear band propagation for further enhancement of the mechanical properties. The breakthrough of amorphous multilayer (ML) film synthesis with bi-layer period down from 200 to 5 nm will allow us to correlate interfacial effects on the mechanical properties in all-amorphous TFMGs (Fig. 12a). In collaboration with the MA department, we used a new strategy to develop thermally stable, ultrastrong and deformable crystal-glass nanocomposites through a thermodynamically guided alloy design approach. We realized this in form of a model CrCoNi (crystalline)/TiZrNbHf (amorphous) nanolaminate composite alloy with bilayer period of 5 nm. This unique nanoarchitecture shows an ultrahigh compressive yield strength of 3.6 GPa and large homogeneous deformation to ~15% strain, surpassing those of conventional metallic glasses and nanolaminates while exhibiting a high crystallization temperature >973 K.

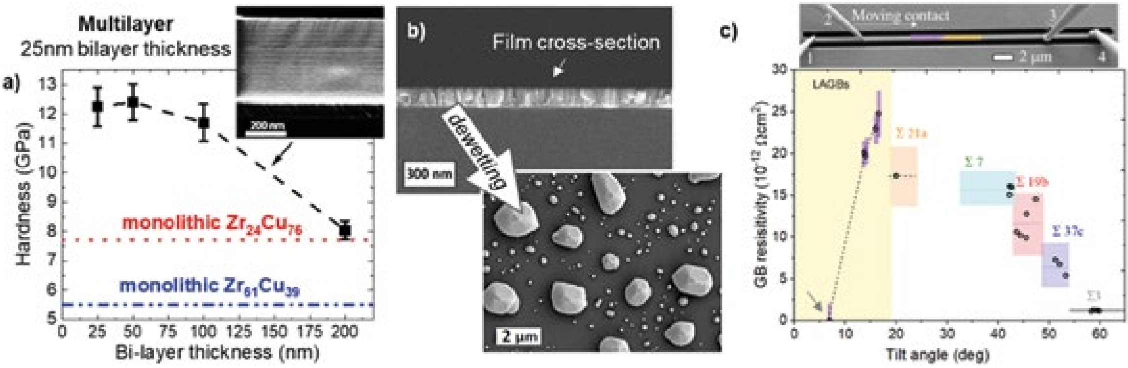


Fig. 12: *Nanoindentation hardness for single and ML ZrCu TFMGs (a). The hardness of MLs with bi-layer thickness of 25 nm (inset, SEM cross-section image) is higher than the rule of mixture for the two components, reaching ~12 GPa. SEM cross-section image of Co-Cu film (~15 at.% Co) showing a densely packed columnar structure (b), together with image after SSD highlighting islands formed after annealing. Microscale in situ electrical resistivity measurements (c) show that the resistivity of specific GBs in Cu varies with tilt angle [16].*

Thin film metallic glasses and nanolayer engineering

Amorphous thin film metallic glass (TFMG) alloys are exciting candidates for next-generation applications, such as flexible electronics, due to their advantageous mechanical properties such as combined high yield strength (up to 3 GPa) and ductility (up to 10 %) [111, 112]. However, the relationships between film chemistry, microstructure and mechanical properties have barely been investigated in this materials class. To address this, we have deposited $Zr_{100-x}Cu_x$ TFMGs with controlled composition by magnetron sputtering, and linked the atomic structure (chemistry) with mechanical properties and thermal stability using nanoindentation, surface Brillouin spectroscopy,

Electrical characteristics of thin film systems

Together with the mechanical performance, the investigation of electrical properties constitutes a major focus, linking to electronic applications such as metallic interconnects or flexible electronics. In the group, we focus this study onto defects in metallic alloys and oxides with special attention on GB engineering. Thin films are deposited by sputtering and then annealed, enabling the production of columnar GB segments which can be investigated at micron and sub-micron scales by *in situ* SEM resistivity measurements developed in-house [1]. Among our achievements is the attainment of abnormal grain growth for epitaxial pure metal (Cu, Ti, Ni and Al) thin films. Moreover, we have enabled the synthesis of desired GB types, whose local electrical properties have been investigated (Fig. 12c) and compared to MD simulations of GB structure performed by T. Brink in the SN department [16]. On-going research involves elucidating the effects of chemical segregation on local resistivity, and in working towards this we have developed a deposition methodology to obtain abnormal growth in Fe-diluted Cu alloys overcoming the pinning of GBs by segregation.

Intermetallic Materials (M. Palm, F. Stein)

Group mission

The investigation of fundamental aspects of the thermodynamic stability of intermetallic phases in view of their possible use for structural applications is the mission of the group Intermetallic Materials.

TiAl-based alloys

Aligned with our group mission, the large-scale international collaborative project ADVANCE (https://thermocalc.com/about-us/advance/) aims at improving a thermodynamic database for the computer-driven development of TiAl-based alloys (see p. 89). In all, the constitution of ten Ti–Al–X(–Y) (X = Nb, Mo, W, O, B, Zr, C, Si; X/Y = Nb/W, Nb/Mo) systems will be investigated. The investigations focus on establishing the phase equilibria and phase transformations in the Ti-Al-rich parts of the systems between 700 – 1400 °C.

Ti–Al–Nb/Mo/W: Currently applied TiAl-based alloys are multicomponent materials with Nb as a major alloying component. Consequently, a detailed knowledge of the phase relations in the ternary Ti–Al–Nb system is of utmost importance. Within the ADVANCE project, the complex ternary phase diagram of this system is constructed by combining results from equilibrated alloys and diffusion couples in the temperature range 700 – 1300 °C [73]. Special care has to be taken regarding the oxygen impurity contents, which were proven to affect the phase equilibria [38]. Another issue is the occurrence of two ternary intermetallic phases (so-called ω0 and O-phase) in the Ti–Al–Nb system, which decompose in the solid state near the regime of desired application temperatures [113, 114]. Moreover, the effect of partial replacement of Nb by Mo or W and the phase diagrams of the ternary systems Ti–Al–Mo and Ti–Al–W are currently under investigation. A comprehensive assessment of the Ti–Al–Mo ternary system has already been performed [78], and existing phase diagram descriptions of the Al–Mo and Ti–Al–W systems were updated [76, 115].

Ti–Al–Si: In this system five partial isothermal sections between 800 – 1200 °C have been established. On equilibrated alloys the phases have been identified by X-ray diffraction (XRD), compositions of the coexisting phases were established by electron probe microanalysis (EPMA) and temperatures of phase transformations were determined by differential thermal analysis [77]. Consistency of the isothermal sections was checked by setting up a vertical section for 9 at.% Si.

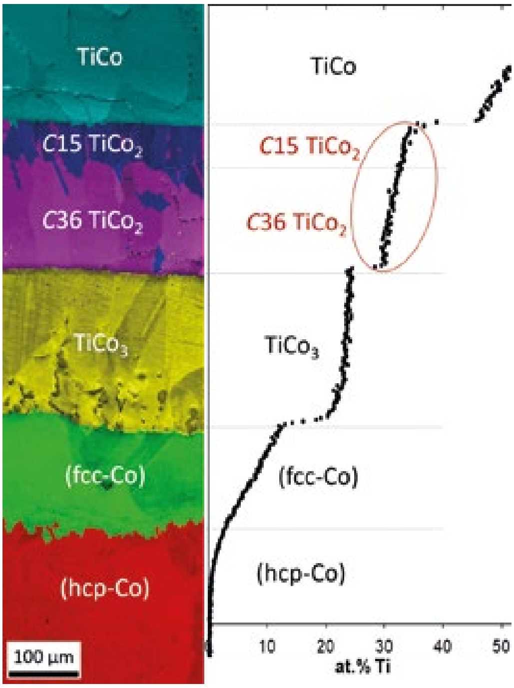


Fig. 13: *EBSD phase map of a Co-TiCo diffusion couple (144 h at 1100 °C) and respective concentration profile revealing the co-existence of C15- and C36-TiCo2 Laves phase without a step in the concentration curve [117].*

Ti–Al–Zr: Investigation of this system started with a critical assessment of the published literature [42]. The ongoing investigations already revealed that phase equilibria are actually quite different from previously published ones [74]. The underlying reason is that phases had been taken into account which are stabilized by impurities. This became apparent as in the present investigation high-purity materials are used and the impurity content is minimized by respective measures during heat treatments and controlled by wet-chemical analysis after annealing [74].

Ti–Al–O: Work on this system again started with a critical assessment of the existing literature [submitted]. As it is a key system not only for TiAl-based alloys, but also for Al_2TiO_5 ceramics, a large number of references are available for this system, which had not been assessed since more than 20 years.

Finally, a critical assessment of the Ti–Al binary system has been updated [116].

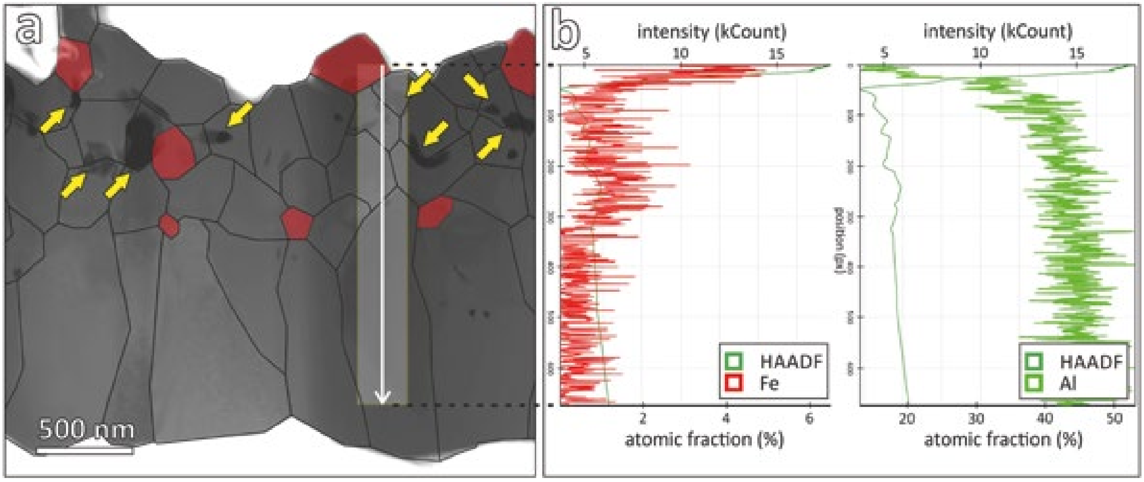


Fig. 14: *a) High-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) micrograph with marked b) STEM energy-dispersive spectrometry (EDS) line scan of Fe-Kα and Al-Kα calculated as atomic fractions, showing the presence of Fe in the upper layer of the scale, while pillar-like alumina at the bottom is pure* Al_2O_3*. To better visualise the grain structure, grain boundaries are marked by lines, and spinel grains have been coloured red. The black patches in the underlying STEM micrograph are corrosion pits (marked by yellow arrows) [49].*

Laves phases

Another research focus of the *Intermetallics group* is on the thermodynamic stability and properties of Laves phases, which form one of the largest groups of intermetallic phases and can exist with three different crystal structure variants (cubic C15, hexagonal C14 and hexagonal C36). In several systems, two or even all three variants can co-exist as equilibrium phases in adjacent composition ranges. An example is the co-existence of C15-$TiCo_2$ and C36-$TiCo_2$ Laves phases in the Co–Ti system [33, 117] (Fig. 13). The phase fields of these two polytypes should be separated by a two-phase field as there is no crystallographic group-subgroup relation between the space groups of the cubic C15 and hexagonal C36 structure type. However, the concentration profile measured on a Co/TiCo diffusion couple (Fig. 13) does not indicate any step-change in composition. A possible explanation might be the presence of a coherent equilibrium between the two structurally closely related phases [117]. Other Laves phase related projects of the group concentrated on the composition and structure dependence of mechanical properties of the C14, C15, and C36 polytype of the $NbCo_2$ Laves phase which exists in an extended homogeneity range of about 10 at.% [40, 41, 118], on the occurrence of extended planar faults in off-stoichiometric $NbFe_2$ Laves phase (where preliminary results were already mentioned in the last Scientific Report) [37], and on the special deformation behaviour of the mono-clinic phase Nb_2Co_7 which possesses a Laves phase related crystal structure [34, 36, 45]. Finally, the group was invited to write a comprehensive overview on Laves phases, which was published in 2021 [48].

Iron aluminides

One of the long-standing research activities at the MPIE is the investigation of fundamental aspects for the development of sustainable iron aluminide alloys. In the framework of a large-scale industrial cooperation devoted to the development and processing of advanced iron aluminide alloys [44, 119, 120], specifically the metastable precipitation of the Heusler phase which is decisive for fine-tuning the microstructure, and has been investigated by atom probe tomography (see p. 186).

Their outstanding corrosion resistance is one of the features that makes iron aluminides so sustainable. However, their aqueous corrosion behaviour is only mediocre and whether it can be improved through pre-oxidation has been controversially discussed in literature due to conflicting experimental evidence. Therefore the aqueous corrosion behaviour of pre-oxidized Fe-25 at.% Al has been investigated in sulphuric acid [121, 122]. The results proved that the oxide scale not only protected the iron aluminide perfectly against aqueous corrosion, but even self-healing of the scale was observed after long-term immersion. To further elucidate why the oxide scale is so protective, it has been analysed using high-resolution techniques [49, 123]. Grazing incident XRD revealed that the scale consists mainly out of α-Al2O3, with a small fraction of spinel ($FeAl_2O_4$). Investigation of a cross section of the immersed oxide scale by TEM methods revealed its microstructure in detail (Fig. 14). Corrosive attack is limited to the outer layer of the scale, which consists of α-Al_2O_3 + $FeAl_2O_4$. The inner layer is formed of columnar α-Al_2O_3 with few grain boundaries, which inhibit further ingress of the acid to the scale/metal interface (Fig. 14). Compressive stresses, which developed during cooling from pre-oxidation at 1000 °C, are also helpful for "sealing" the grain boundaries [49].

Other activities on iron aluminides and iron-based alloys include critical assessments of the phase equilibria, phase transformations and thermodynamics of the ternary systems Fe–Al–Co [75], Fe–Nb–Ti [124], and Fe–Nb–V [125], a summarizing account of a collaboration with industries on the characterization of the microstructure, corrosion and mechanical behaviour of combustion chamber prototypes for marine engines before and after testing [126], and investigations by electron probe micro analysis (EPMA) to settle whether cementite (Fe_3C) shows a homogeneity range [127].

The importance of iron aluminides as a new class of sustainable materials for CO_2-reduced transportation and energy applications, and the substantial contributions at the MPIE for their development, have been demonstrated in a couple of honourable publications. An invited review on the history, phase diagram and crystal structures, physical and mechanical properties, corrosion behaviour, synthesis, processing and application has been published [32]. A discussion of the role of iron aluminides as inexpensive and easily recyclable alloys for demanding applications was chosen by the Max Planck Society as one of the 'Highlights from the yearbook of the Max Planck Society 2019' [128], and there has been a radio Deutschlandfunk report about the group's work on sustainable iron aluminides [129]. Iron aluminides were also included in a book chapter on 'Iron-Based Intermetallics', which in addition dealt with iron silicides, iron-based ferritic superalloys, and iron-based alloys with TCP phases [46]. Finally, a publication contract to edit a book on 'Ternary Iron Phase Diagrams Relevant for the Application of Iron Aluminides' has been signed.

References

1. Bishara, H.; Ghidelli, M.; Dehm, G.: ACS Appl. Electron. Mater. 2 (2020) 2049.
2. Brinckmann, S.; Soler, R.; Dehm, G.: Materialia 8 (2019) 100421.
3. Arigela, V.G.; Oellers, T.; Ludwig, A.; Kirchlechner, C.; Dehm, G.: Rev. Sci. Instrum. 90 (2019) 073904.
4. Oellers, T.; Arigela, V.G.; Kirchlechner, C.; Dehm, G.; Ludwig, A.: ACS Comb. Sci. 22 (2020) 142 .
5. Saxena, A.K.; Brinckmann, S.; Völker, B.; Dehm, G.; Kirchlechner, C.: Mater. Des. 191 (2020) 108582.
6. Seok, M.-Y.; Gopalan, H.; Nandy, S.; Zaefferer, S.; Raabe, D.; Kirchlechner, C.; Dehm, G.: Materialia 14 (2020) 100932.
7. Duarte, M.J.; Fang, X.; Rao, J.; Krieger, W.; Brinckmann, S.; Dehm, G.: J. Mater. Sci. 56 (2021) 8732 .
8. Ramachandramoorthy, R.; Yang, F.; Casari, D.; Stolpe, M.; Jain, M.; Schwiedrzik, J.; Michler, J.; Kruzic, J.J.; Best, J.P.: J. Mater. Res. 36 (2021) 2325.
9. Malyar, N.V.; Grabowski, B.; Dehm, G.; Kirchlechner, C.: Acta Mater. 161 (2018) 412 .
10. Kini, M.K.; Dehm, G.; Kirchlechner, C.: Acta Mater. 184 (2020) 120 .
11. Ko, W.-S.; Stukowski, A.; Hadian, R.; Nematollahi, G.A.; Jeon, J.B.; Choi, W.S.; Dehm, G.; Neugebauer, J.; Kirchlechner, C.; Grabowski, B.: Acta Mater. 197 (2020) 54 .
12. Lee, S.; Duarte, M.J.; Feuerbacher, M.; Soler, R.; Kirchlechner, C.; Liebscher, C.; Oh, S.H.; Dehm, G.: Mater. Res. Lett. 8 (2020) 216 .
13. Utt, D.; Lee, S.; Stukowski, A.; Oh, S.H.; Dehm, G.; Albe, K.: ArXiv 2007.11489 (2020).
14. Fang, X.; Kreter, A.; Rasiński, M.; Kirchlechner, C.; Brinckmann, S.; Linsmeier, C.; Dehm, G.: J. Mater. Res. 33 (2018) 3530 .
15. Fang, X.; Rasiński, M.; Kreter, A.; Kirchlechner, C.; Linsmeier, C.; Dehm, G.; Brinckmann, S.: Scr. Mater. 162 (2019) 132 .
16. Bishara, H.; Lee, S.; Brink, T.; Ghidelli, M.; Dehm, G.: ACS Nano 15 (2021) 16607.
17. Minor, A.M.; Dehm, G.: MRS Bulletin 44 (2019) 438 .
18. Guo, J.; Duarte, M.J.; Zhang, Y.; Bachmaier, A.; Gammer, C.; Dehm, G.; Pippan, R.; Zhang, Z.: Acta Mater. 166 (2019) 281 .
19. Zhao, H.; Huber, L.; Lu, W.; Peter, N.J.; An, D.; De Geuser, F.; Dehm, G.; Ponge, D.; Neugebauer, J.; Gault, B.; Raabe, D.: Phys. Rev. Lett. 124 (2020) 106102.
20. Wu, X.; Makineni, S.K.; Liebscher, C.; Dehm, G.; Mianroodi, J.R.; Shanthraj, P.; Svendsen, B.; Bürger, D.; Eggeler, G.F.; Raabe, D.; Gault, B.; Gault, B.: Nat. Commun. 11 (2020) 389.
21. Lu, W.; Liebscher, C.; Yan, F.; Fang, X.; Li, L.; Li, J.; Guo, W.; Dehm, G.; Raabe, D.; Li, Z.: Acta Mater. 185 (2020) 218 .
22. Meiners, T.; Duarte, M.J.; Richter, G.; Dehm, G.; Liebscher, C.: Acta Mater. 190 (2020) 93 .
23. Meiners, T.; Frolov, T.; Rudd, R.E.; Dehm, G.; Liebscher, C.: Nature 579 (2020) 375 .
24. Elhebeary, M.; Harzer, T.; Dehm, G.; A. Saif, M.T.: Proc. Natl. Acad. Sci. 117 (2020) 16864.
25. Chakraborty, J.; Harzer, T.P.; Duarte, M.J.; Dehm, G.: J. Alloys Compd. 888 (2021) 161391.
26. Ahmadian, A.; Scheiber, D.; Zhou, X.; Gault, B.; Liebscher, C.; Romaner, L.; Dehm, G.: Nat. Commun. 12 (2021) 6008.
27. Frommeyer, L.; Brink, T.; Freitas, R.; Frolov, T.; Dehm, G.; Liebscher, C.H.: ArXiv 2109.15192 (2021).
28. Peter, N.J.; Duarte, M.J.; Kirchlechner, C.; Liebscher, C.; Dehm, G.: Acta Mater. 214 (2021) 116960.
29. Evertz, S.; Kirchlechner, I.; Soler, R.; Kirchlechner, C.; Kontis, P.; Bednarcik, J.; Gault, B.; Dehm, G.; Raabe, D.; Schneider, J.M.: Mater. Des. 186 (2020) 108327.
30. Jeong, J.; Cautaerts, N.; Dehm, G.; Liebscher, C.H.: Microsc. Microanal. 27 (2021) 1102.
31. Chauniyal, A.; Dehm, G.; Janisch, R.: J. Mech. Phys. Solids 154 (2021) 104511.
32. Palm, M.; Stein, F.; Dehm, G.: Annu. Rev. Mater. Res. 49 (2019) 297.
33. Stein, F.; Merali, M.; Watermeyer, P.: Proc. Intermetallics 2019 (2019) 55.
34. Horiuchi, T.; Yamada, K.; Ikeda, K.-I.; Miura, S.; Stein, F.: Proc. Intermetallics 2019 (2019) 135.
35. Šestan, A.; Zavašnik, J.; Kržmanc, M.M.; Kocen, M.; Jenuš, P.; Novak, S.; Čeh, M.S.; Dehm, G.: J. Nucl. Mater. 524 (2019) 135.

36. Yamada, K.; Horiuchi, T.; Miura, S.; Stein, F.: Proc. Intermetallics 2019 (2019) 210.
37. Šlapáková, M.; Zendegani, A.; Liebscher, C.H.; Hickel, T.; Neugebauer, J.; Hammerschmidt, T.; Ormeci, A.; Grin, J.; Dehm, G.; Kumar, K.S.; Stein, F.: Acta Mater. 183 (2020) 362.
38. Distl, B.; Dehm, G.; Stein, F.: Z. Anorg. Allg. Chem. 646 (2020) 1151.
39. Laursen, C.M.; Peter, N.J.; Gerstein, G.; Maier, H.J.; Dehm, G.; Frick, C.P.: Mater. Sci. Eng. A 792 (2020) 139373.
40. Luo, W.; Kirchlechner, C.; Zavašnik, J.; Lu, W.; Dehm, G.; Stein, F.: Acta Mater. 184 (2020) 151.
41. Luo, W.; Kirchlechner, C.; Li, J.; Dehm, G.; Stein, F.: J. Mater. Res. 35 (2020) 185.
42. Kahrobaee, Z.; Palm, M.: J. Phase Equilib. Diffus. 41 (2020) 687.
43. Cautaerts, N.; Rauch, E.F.; Jeong, J.; Dehm, G.; Liebscher, C.: Scr. Mater. 201 (2021) 113930.
44. Gedsun, A.; Stein, F.; Palm, M.: MRS Adv. 6 (2021) 176.
45. Horiuchi, T.; Yamaguchi, M.; Yamada, K.; Ikeda, K.-I.; Miura, S.; Stein, F.: Proc. Intermetallics 2021 (2021) 51.
46. Palm, M.; Stein, F.: Iron-Based Intermetallics, in: Rana, R. (ed.), High-Performance Ferrous Alloys, Springer Nature Switzerland AG, Cham, Switzerland (2021) 423.
47. Rehman, U.; Tian, C.; Stein, F.; Best, J.P.; Dehm, G.: Proc. Intermetallics 2021 (2021) 155.
48. Stein, F.; Leineweber, A.: J. Mater. Sci. 56 (2021) 5321.
49. Zavašnik, J.; Peng, J.; Palm, M.: Corros. Sci. 179 (2021) 109170.
50. Guo, W.; Su, J.; Lu, W.; Liebscher, C.; Kirchlechner, C.; Ikeda, Y.; Körmann, F.; Liu, X.; Xue, Y.; Dehm, G.: Acta Mater. 185 (2020) 45.
51. Jung, H.Y.; Peter, N.J.; Gärtner, E.; Dehm, G.; Uhlenwinkel, V.; Jägle, E.A.: Addit. Manuf. 35 (2020) 101337.
52. Lee, S.; Chatain, D.; Liebscher, C.; Dehm, G.: Scr. Mater. 203 (2021) 114044.
53. Wolff-Goodrich, S.; Haas, S.; Glatzel, U.; Liebscher, C.: Acta Mater. 216 (2021) 117113.
54. Wolff-Goodrich, S.; Marshal, A.; Pradeep, K.G.; Dehm, G.; Schneider, J.M.; Liebscher, C.: J. Alloys Compd. 853 (2021) 156111.
55. Cao, T.; Guo, W.; Lu, W.; Xue, Y.; Lu, W.; Su, J.; Liebscher, C.H.; Li, C.; Dehm, G.: J. Alloys Compd. 891 (2022) 161859.
56. Rogal, Ł.; Morgiel, J.; Stein, F.; Breitbach, B.; Dutkiewicz, J.: Mater. Charact. 148 (2019) 134.
57. Addab, Y.; Kini, M.K.; Courtois, B.; Savan, A.; Ludwig, A.; Bozzolo, N.; Scheu, C.; Dehm, G.; Chatain, D.: Acta Mater. 200 (2020) 908.
58. Kini, M.K.; Lee, S.; Savan, A.; Breitbach, B.; Addab, Y.; Lu, W.; Ghidelli, M.; Ludwig, A.; Bozzolo, N.; Scheu, C.; Chatain, D.; Dehm, G.: Surf. Coat. Technol. 410 (2021) 126945.
59. Devulapalli, V.; Bishara, H.; Ghidelli, M.; Dehm, G.; Liebscher, C.: Appl. Surf. Sci. 562 (2021) 150194.
60. Du, C.; Soler, R.; Völker, B.; Matoy, K.; Zechner, J.; Langer, G.; Reisinger, M.; Todt, J.; Kirchlechner, C.; Dehm, G.: Materialia 8 (2019) 100503.
61. Evertz, S.; Schnabel, V.; Köhler, M.; Kirchlechner, I.; Kontis, P.; Chen, Y.-T.; Soler, R.; Jaya, B.N.; Kirchlechner, C.; Mušić, D.; Gault, B.; Gault, B.; Schneider, J.M.; Raabe, D.; Dehm, G.: Front. Mater. 7 (2020) 89.
62. Glushko, O.; Dehm, G.: Acta Mater. 169 (2019) 99 .
63. Hieke, S.W.; Willinger, M.G.; Wang, Z.-J.; Richter, G.; Chatain, D.; Dehm, G.; Scheu, C.: Acta Mater. 165 (2019) 153 .
64. Kini, M.K.; Merola, C.; Breitbach, B.; Klapproth, D.; Philippi, B.; Molin, J.-B.; Kirchlechner, C.; Dehm, G.: Acta Mater. 198 (2020) 61.
65. Liu, C.; Li, Z.; Lu, W.; Bao, Y.; Xia, W.; Wu, X.; Zhao, H.; Gault, B.; Liu, C.; Herbig, M.; Fischer, A.; Dehm, G.; Wu, G.; Raabe, D.: Nat. Commun. 12 (2021) 5518.
66. Marshal, A.; Singh, P.; Mušić, D.; Wolff-Goodrich, S.; Evertz, S.; Schökel, A.; Johnson, D.D.; Dehm, G.; Liebscher, C.; Schneider, J.M.: J. Alloys Compd. 854 (2021) 155178.
67. Völker, B.; Stelzer, B.; Mráz, S.; Rueß, H.; Sahu, R.; Kirchlechner, C.; Dehm, G.; Schneider, J.M.: Mater. Des. 206 (2021) 109757.
68. Zhang, Z.; Chen, Z.; Holec, D.; Liebscher, C.; Koutná, N.; Bartosik, M.; Zheng, Y.; Dehm, G.; Mayrhofer, P.H.: Acta Mater. 194 (2020) 343 .
69. Wu, G.; Balachandran, S.; Gault, B.; Xia, W.; Liu, C.; Rao, Z.; Wei, Y.; Liu, S.; Lu, J.; Herbig, M.; Lu, W.; Dehm, G.; Li, Z.; Raabe, D.: Adv. Mater. 32 (2020) 2002619.
70. Malyar, N.V.; Springer, H.; Wichert, J.; Dehm, G.; Kirchlechner, C.: Mater. Test. 61 (2019) 5 .
71. Garzón-Manjón, A.; Christiansen, L.; Kirchlechner, C.; Breitbach, B.; Liebscher, C.; Springer, H.; Dehm, G.: J. Alloys Compd. 794 (2019) 203 .
72. Wolff-Goodrich, S.: Development of AlCrFeNiTi compositionally complex alloys for high temperature structural applications, Doctoral Thesis, Ruhr-Universität Bochum (2021).
73. Distl, B.; Palm, M.; Stein, F.; Rackel, M.; Hauschildt, K.; Pyczak, F.: Proc. Intermetallics 2019 (2019) 170.
74. Kahrobaee, Z.; Stein, F.; Palm, M.: Proc. Intermetallics 2019 (2019) 174.
75. Kumar, H.K.C.; Palm, M.; Rank, M.; Walnsch, A.; Watson, A.: Aluminium – Cobalt – Iron, in: Effenberg, G. (ed.), Ternary Phase Diagrams, Materials Science International Services GmbH, Stuttgart (2019) 10.15955.3.7.
76. Hayes, F.; Distl, B.: Aluminium – Titanium – Tungsten, in: Watson, A. (ed.), Ternary Phase Diagrams, Materials Science International Services GmbH, Stuttgart, Germany (2020) 10.22122.2.4.
77. Kahrobaee, Z.; Palm, M.: Proc. Intermetallics 2021 (2021) 78.
78. Distl, B.; Walnsch, A.; Mellor, R.F.L.; Gomell, L.; Noori, M.; Gedsun, A.; Stein, F.: Aluminium – Molybdenum – Titanium, in: Watson, A. (ed.), Ternary Phase Diagrams, Materials Science International Services GmbH, Stuttgart, Germany (2021) 10.17143.3.2.
79. Lu, W.; Liebscher, C.; Dehm, G.; Raabe, D.; Li, Z.: Adv. Mater. 30 (2018).
80. Zelenina, I.; Simon, P.; Veremchuk, I.; Wang, X.; Bobnar, M.; Lu, W.; Liebscher, C.H.; Grin, Y.: Commun. Mater. 2 (2021) 99.
81. Berkels, B.; Liebscher, C.: Ultramicroscopy 198 (2019) 49 .
82. Wang, N.; Freysoldt, C.; Zhang, S.; Liebscher, C.H.; Neugebauer, J.: Microsc. Microanal. (2021) 1.
83. Peter, N.J.; Frolov, T.; Duarte, M.J.; Hadian, R.; Ophus, C.; Kirchlechner, C.; Liebscher, C.; Dehm, G.: Phys. Rev. Lett. 121 (2018).
84. Chen, H.; Kauffmann, A.; Seils, S.; Boll, T.; Liebscher, C.; Harding, I.; Kumar, K.S.; Szabó, D.V.; Schlabach, S.; Kauffmann-Weiss, S.; Müller, F.E.H.; Gorr, B.; Christ, H.-J.; Heilmaier, M.: Acta Mater. 176 (2019) 123 .
85. Wang, Z.; Lu, W.; Zhao, H.; Liebscher Christian, H.; He, J.; Ponge, D.; Raabe, D.; Li, Z.: Sci. Adv. 6 (2020) eaba9543.

86. Vasileiadis, I.G.; Lymperakis, L.; Adikimenakis, L.A.; Gkotinakos, A.; Devulapalli, V.; Liebscher, C.H.; Androulidaki, M.; Hübner, R.; Karakostas, T.; Georgakilas, A.; Komninou, P.; Dimakis, E.; Dimitrakopulos, G.P.: Sci. Rep. 11 (2021) 20606.
87. Liebscher, C.; Stoffers, A.; Alam, M.; Lymperakis, L.; Cojocaru-Mirédin, O.; Gault, B.; Neugebauer, J.; Dehm, G.; Scheu, C.; Raabe, D.: Phys. Rev. Lett. 121 (2018) 015702.
88. Raghavan, R.; Kirchlechner, C.; Jaya, B.N.; Feuerbacher, M.; Dehm, G.: Scr. Mater. 129 (2017) 52.
89. Tian, C.; Ponge, D.; Christiansen, L.; Kirchlechner, C.: Acta Mater. 183 (2020) 274 .
90. Tian, C.; Dehm, G.; Kirchlechner, C.: Materialia 15 (2021) 100983.
91. Li, J.; Kirchlechner, C.: Mater. Charact. 161 (2020) 110136.
92. Li, J.; Pharr, G.M.; Kirchlechner, C.: J. Mater. Res. 36 (2021) 2037.
93. Dehm, G.; Jaya, B.N.; Raghavan, R.; Kirchlechner, C.: Acta Mater. 142 (2018) 248 .
94. Gopalan, H.; Amalraj, M.; Völker, B.; Kirchlechner, C.; Dehm, G.; Schneider, J.M.: in: Molina-Aldareguia, J. (ed.), Nanomechanical Testing in Materials Research and Development VII, ECI Symposium Series, Malaga, Spain, (2019).
95. Völker, B.; Du, C.; Fager, H.; Rueß, H.; Soler, R.; Kirchlechner, C.; Dehm, G.; Schneider, J.M.: Surf. Coat. Technol. 390 (2020) 125645.
96. Tian, C.; Kirchlechner, C.: J. Mater. Res. 36 (2021) 2495 .
97. Kumar, A.; Saxena, A.K.; Kirchlechner, C.; Herbig, M.; Brinckmann, S.; Petrov, R.; Sietsma, J.: Acta Mater. 180 (2019) 60 .
98. Saxena, A.K.; Kumar, A.; Herbig, M.; Brinckmann, S.; Dehm, G.; Kirchlechner, C.: Mater. Des. 180 (2019) 107892.
99. Mathews, N.G.; Saxena, A.K.; Kirchlechner, C.; Dehm, G.; Jaya, B.N.: Scr. Mater. 182 (2020) 68 .
100. Wu, Y.Q.; Gao, S.; Kang, R.K.; Huang, H.: J. Mater. Sci. 54 (2019) 1958.
101. Fang, X.; Bishara, H.; Ding, K.; Tsybenko, H.; Porz, L.; Höfling, M.; Bruder, E.; Li, Y.; Dehm, G.; Durst, K.: J. Am. Ceram. Soc. 104 (2021) 4728 .
102. Ramachandramoorthy, R.; Schwiedrzik, J.; Petho, L.; Guerra-Nunez, C.; Frey, D.; Breguet, J.-M.; Michler, J.: Nano letters 19 (2019) 2350.
103. Peruzzi, C.; Ramachandramoorthy, R.; Groetsch, A.; Casari, D.; Grönquist, P.; Rüggeberg, M.; Michler, J.; Schwiedrzik, J.: Acta Biomater. 131 (2021) 403.
104. Ramachandramoorthy, R.; Milan, M.; Lin, Z.; Trolier-McKinstry, S.; Corigliano, A.; Espinosa, H.: Extreme Mech. Lett. 20 (2018) 14.
105. Schürch, P.; Ramachandramoorthy, R.; Pethö, L.; Michler, J.; Philippe, L.: Appl. Mater. Today 18 (2020) 100472.
106. Xia, W.; Dehm, G.; Brinckmann, S.: Wear 452–453 (2020) 203289.
107. Xia, W.; Dehm, G.; Brinckmann, S.: Mater. Des. 183 (2019) 108169.
108. Xia, W.; Dehm, G.; Brinckmann, S.: J. Mater. Sci. 55 (2020) 9095.
109. Tsybenko, H.; Xia, W.; Dehm, G.; Brinckmann, S.: Tribol. Int. 151 (2020) 106477.
110. Tsybenko, H.; Farzam, F.; Dehm, G.; Brinckmann, S.: Tribol. Int. 163 (2021) 107168.
111. Ghidelli, M.; Idrissi, H.; Gravier, S.; Blandin, J.-J.; Raskin, J.-P.; Schryvers, D.; Pardoen, T.: Acta Mater. 131 (2017) 246.
112. Ghidelli, M.; Orekhov, A.; Bassi, A.L.; Terraneo, G.; Djemia, P.; Abadias, G.; Nord, M.; Béché, A.; Gauquelin, N.; Verbeeck, J.; Raskin, J.P.; Schryvers, D.; Pardoen, T.; Idrissi, H.: Acta Mater. 213 (2021) 116955.
113. Distl, B.; Hauschildt, K.; Pyczak, F.; Stein, F.: Metals 11 (2021) 1991.
114. Distl, B.; Stein, F.: Proc. Intermetallics 2021 (2021) 67.
115. Walnsch, A.; Stein, F.: Aluminium–Molybdenum, in: Watson, A. (ed.), Binary Phase Diagrams, Materials Science International Services GmbH, Stuttgart, Germany (2020) 20.12123.3.1.
116. Palm, M.: Aluminium–Titanium, in: Effenberg, G. (ed.), Binary Phase Diagrams, Materials Science International Services GmbH, Stuttgart (2020) 1.
117. Stein, F.; Merali, M.; Watermeyer, P.: Calphad 67 (2019) 101681.
118. Luo, W.; Kirchlechner, C.; Dehm, G.; Stein, F.: in: Shingledecker, J.; Takeyama, M. (ed.), Proc. Joint EPRI – 123HiMAT International Conference on Advances in High Temperature Materials, Nagasaki, Japan, ASM International, Materials Park, OH, USA (2019) 11.
119. Gedsun, A.; Palm, M.: Proc. Intermetallics 2019 (2019) 208.
120. Gedsun, A.; Palm, M.: Proc. Intermetallics 2021 (2021) 121.
121. Peng, J.; Moszner, F.; Rechmann, J.; Vogel, D.; Palm, M.; Rohwerder, M.: Corros. Sci. 149 (2019) 123.
122. Zavašnik, J.; Peng, J.; Palm, M.: Proc. Intermetallics 2019 (2019) 70.
123. Zavašnik, J.; Peng, J.; Palm, M.: in: Godec, M.; Donik, Č.; Kocijan, A., Paulin, I. (eds.), 27. Int. Conf. Mater. Technol., Portorož, Slovenija, Inštitut za Kovinske Materiale in Tehnologije, Ljubiljana, (2019) 187.
124. Kriegel, M.J.; Fels, J.; Stein, F.: Iron–Niobium–Titanium, in: Effenberg, G. (ed.), Ternary Phase Diagrams, Materials Science International Services GmbH, Stuttgart (2019) 10.36946.1.5.
125. Dreval, L.; Distl, B.; Hübner, J.-M.; Bueno Villoro, R.; Kahrobaee, Z.; Schmitt, L.-Y.; Iljenko, S.; Shiriaev, V.; Prokopenko, R.: Iron–Niobium–Vanadium, in: Effenberg, G. (ed.), Ternary Phase Diagrams, Materials Science International Services GmbH, Stuttgart, Germany (2019) 10.38583.1.0.
126. Moszner, F.; Peng, J.; Suutala, J.; Jasnau, U.; Damani, M.; Palm, M.: Metals 9 (2019) 847.
127. Schneider, A.; Palm, M.: Calphad 68 (2020) 101689.
128. Palm, M.; Stein, F.; Gedsun, A.; Dehm, G.: Sustainable Alloys for Demanding Applications, in: (ed.), Highlights from the Yearbook of the Max Planck Society 2019, Max Planck Society, Munich, Germany (2020) 38.
129. Mrasek, V.: Der Stahl der Zukunft: Neue Legierungen sollen den Werkstoff nachhaltiger machen, Forschung Aktuell – Deutschlandfunk (2021) https://www.deutschlandfunk.de/der-stahl-der-zukunft-neue-legierungen-sollen-den-werkstoff-nachhaltiger-machen-dlf-974f03e8-100.html

Part III.

Interdepartmental research activities – selected highlights

Sustainability of and through materials

Stability of surfaces and interfaces

Microstructure-related material properties

Development of advanced materials

Scale-bridging simulation and materials informatics

Fundamentals of sustainable hydrogen-based metallurgy

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S. Zhang[5], J.R. Mianroodia[1], B. Svendsen[3], Y. Bai[1], X. Zhou[1], C. Scheu[5], D. Raabe[1]

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The massive CO_2 emissions associated with modern iron- and steelmaking have become one of the largest environmental burdens of our generation, and the international steel market is forecast to grow by at least 30–35 % during the next 30 years [1, 2]. Therefore, the institute conducts interdisciplinary and multiscale research on the physical and chemical foundations for improving the sustainability of steels, with a focus on reduced CO_2-intense production and low-energy synthesis. These goals can in principle be reached by combining several types of iron carriers, such as oxidic fines, lump ore, or scrap, with a variety of carbon-free reduction media, such as hydrogen or ammonia, in different types of furnaces.

The interplay of multiple mechanisms and influencing factors both, from the reactive metallurgy side and from the advanced processing side, requires the cooperation of several departments on this topic. This includes the design and operation of fully instrumented reactor set-ups in which the different feedstock materials can be charged, while the underlying redox reactions can be monitored through the spectroscopy of the intermediate and final reaction products.

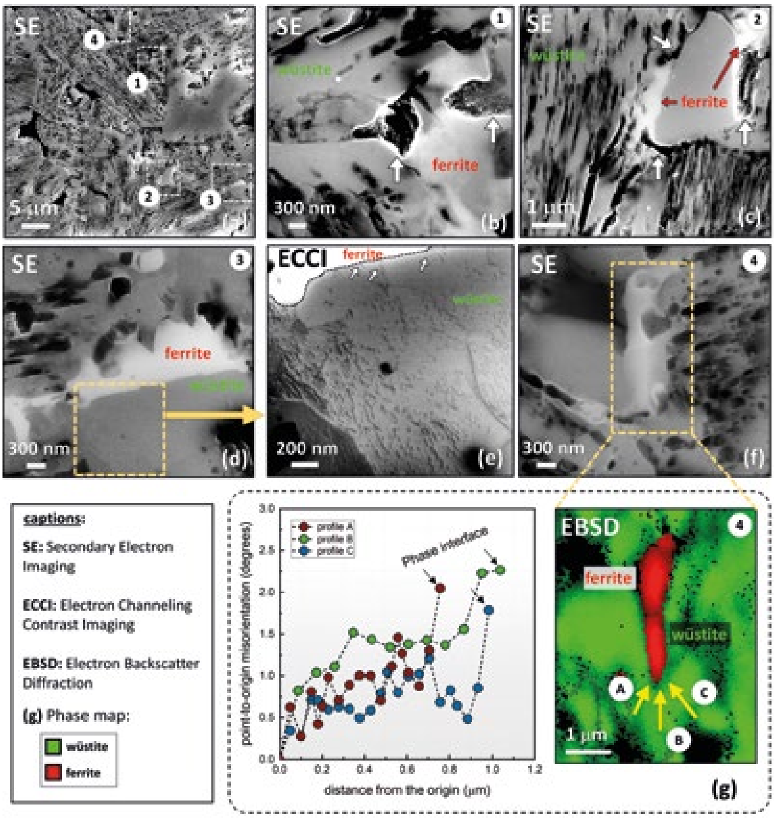

Fig. 1: *Correlative SE and ECCI analysis of a partially reduced pellet (10 min, 700 °C). (a) SE image of the map of the pellet microstructure. The white frames marked as 1, 2, and 3 highlight specific regions whose enlarged views are given in (b), (c), and (d), respectively. (e) ECCI image obtained from the area delimited by the yellow frame in (d). (f) SE image from the area labelled as 4 in (a). The arrows A, B, and C were used as a reference to obtain local orientation gradients. (g) EBSD phase map from the marked area. Point-to-origin local misorientation profiles along arrows A, B, and C in (f).*

The specific focus of this cross-departmental initiative is currently placed on hydrogen-based direct reduction [3] and hydrogen-based plasma smelting reduction of iron ores [4]. As raw materials, commercial pellets and ore fines are used, as well as well-defined oxide single crystals. The hydrogen-based direct reduction of iron oxides is a solid-gas reaction. The kinetics of this process is not well understood, particularly during the wüstite reduction step, which is nearly an order of magnitude slower than the hematite reduction.

The rate-limiting factors of this reaction are investigated and related to the microstructure and local chemistry of the ores, using a multiscale structure and composition analysis of iron reduced from hematite with pure molecular hydrogen reaching down to near-atomic scale [3]. During reduction, a complex pore- and microstructure evolves, due to oxygen loss and several non-volume-conserving phase transformations. The microstructure after reduction is an aggregate of nearly pure iron crystals, containing inherited and acquired pores and cracks (Fig. 1). We observe several types of lattice defects that accelerate mass transport as well as several chemical impurities (Na, Mg, Ti, V) within the Fe in the form of oxide islands that were not reduced.

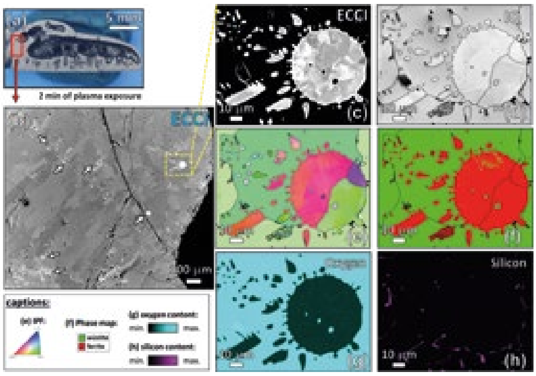

Fig. 2: *Microstructural characterization of a sample partially reduced for 2 min by hydrogen plasma. (a) Overview of the sample. The red frame shows the chosen area for the correlative EBSD-ECCI probing approach. (b) ECCI-image of the area delimited by the red frame in (a). The white arrows evidence the presence of small domains of iron. (c) ECCI-image showing an enlarged view of the region delimitated by the yellow frame in (a). The corresponding EBSD maps of the area displayed in (c) are as follows: (d) image quality (IQ) map; (e) inverse pole figure (IPF) map in which the crystallographic orientations are shown perpendicular to the normal direction of the sample, i.e. parallel to the solidification direction; (f) phase map where wüstite and ferrite are represented by green and red respectively; (g) oxygen distribution and (h) silicon distribution maps.*

Hydrogen-based plasma smelting reduction offers an attractive alternative to green steel production. Therefore, the basic mechanisms behind the reduction of hematite using hydrogen plasma are studied [4]. The reduction kinetics depends on the balance between the input mass and the arc power. For an optimized input mass-arc power ratio, the complete reduction was obtained within 15 min of exposure to the hydrogen plasma. In such a process, wüstite reduction is again the rate-limiting step. Nevertheless, its rates are comparable with those found in hydrogen-based direct reduction. The possible advantage of the reduction of ores through a reductant plasma in an electric arc lies in the fact that reduction and liquification are done in a single process step.

Micro- and nanoscale chemical and microstructure analysis revealed that the gangue elements partition to the remaining oxide regions (Fig. 2). Si-enrichment was observed in the interdendritic fayalite domains, at the wüstite/-iron interfaces, and in the primarily solidified oxide particles inside the iron. With proceeding reduction, however, such elements are gradually removed from the samples so that the final iron product is nearly free of gangue impurities. The studies provide microstructural and atomic-scale insights into the composition and phase transformations during hydrogen-based plasma smelting reduction, enabling better understanding of the underlying thermodynamics and kinetic barriers of this process, as a basis for the design of suited furnace concepts for CO_2-free ironmaking.

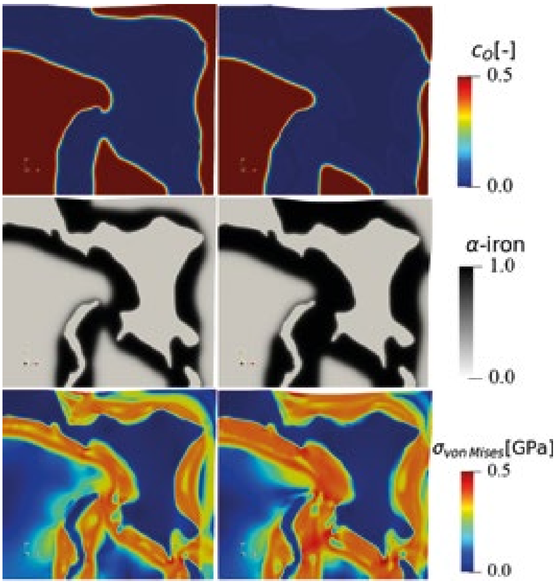

Fig. 3: *Results from the chemo-mechanically coupled phase-field simulations with an elasto-plastic material response for the case of wüstite reduction for 700 °C under hydrogen exposure as a function of reduction time. The images show contour plots of the oxygen concentration, the BCC iron phase order parameter, and the von Mises stress.*

The hydrogen-based direct reduction of iron oxides is also investigated theoretically. A chemo-mechanically coupled phase-field model is currently under development, to explore the interplay between phase transformation, chemical reaction, component diffusion, elastoplastic deformation, and microstructure during the reduction of wüstite with hydrogen (Fig. 3).

The phase-field simulation is coupled to Gibbs free energy data that are fitted from corresponding thermodynamic databases. The simulation reveals the important role of elastic and plastic deformation during the reduction and allows first insights into the different mass transport pathways along the highly defected microstructure. The model also allows to better understand both, the main decelerating factors (oxide-core-metal-shell structures) and accelerating factors (delamination and fracture at the hetero-interfaces) determining the overall reduction kinetics.

References

1. IEA. Iron and Steel Technology: Towards More Sustainable Steelmaking (International Energy Agency, 2020).
2. Pei, M.; Petäjäniemi, M.; Regnell, A.; Wijk, O.: Metals 10 (2020) 972.
3. Kim, S.H.; Zhang, X.; Ma, Y.; Souza Filho, I.R.; Schweinar, K.; Angenendt, K.; Vogel, D.; Stephenson, L.T.; El-Zoka, A.A.; Mianroodi, J.R.; Rohwerder, M.; Gault, B.; Raabe, D.: Acta Mater. 212 (2021) 116933.
4. Souza Filho, I.R.; Ma, Y.; Kulse, M.; Ponge, D.; Gault, B.; Springer, H.; Raabe, D.: Acta Mater. 213 (2021) 116971.

Materials for electrocatalysis

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The world's transition to a more sustainable future requires innovative solutions for energy conversion and storage. Strategies to develop stable, active and durable catalytic materials are an essential part of this development. The continued and concerted effort of different groups at the MPIE to enhance our understanding of the fundamental processes governing the stability, activity and degradation of, e.g., electrolysers in realistic environments and under operando conditions, opens new routes towards designing efficient and long-lived catalysts. Three main directions were pursued within the last years: (i) exploiting metal dealloying to enhance catalytic activity, (ii) understanding the role of dopants, impurities and local inhomogeneities on catalytic activity and (iii) the study of oxides and oxidised compounds, which exhibit an enhanced stability in the conventionally harsh environments developing during catalytic reactions. In all of these, particular attention is paid to resolve the atomistic structure, as the gained insights have proven highly beneficial in explaining catalytic performance.

Our work demonstrated that electrochemical dealloying can greatly improve the catalytic activity of AgAu nanoparticles for the hydrogen evolution reaction [1]. By combining electrochemistry with identical location electron microscopy analyses and linking them to the electrocatalytic properties of the obtained nanocatalysts, they established guidelines for the selection of dealloying parameters to reach highly porous and active materials beyond the previous "trial and error" attempts. Using AgAu alloy nanoparticles and the hydrogen evolution reaction as a model system, the influence of cyclic voltammetry parameters on the catalytic activity upon electrochemical dealloying was investigated. Increasing cycle numbers initially result in a decreased Ag content and a sharp improvement in catalyst activity. Additional dealloying increased the nanoparticle porosity, while marginally altering their composition, due to surface motion of atoms. Since this is accompanied by particle aggregation, further cycling results in a decrease of catalytic activity. This transition between porosity formation and particle aggregation marks the optimum for nanocatalyst post-production. The gained insights will allow speeding up the development of new materials by electrochemical dealloying as an easy-to-control post-processing route to tune properties of existing nanoparticles, instead of having to alter usually delicate synthesis routes as a whole.

Chemical synthesis is a conventional route by which catalytic materials are produced and/or designed. Wet-chemical methods are however, more complex than expected and often reported. Careful analysis of samples we studied in the context of electrocatalysis, revealed the presence of impurities integrated within the structure during synthesis. The presence of traces of such spurious elements may enhance or detrimentally affect the properties of materials and thus, affect their performance. Identification and quantification of such impurities is however extremely challenging. To better tackle these issues, (scanning) transmission electron microscopy ((S)TEM), atom probe tomography (APT) and X-ray photoelectron spectroscopy (XPS) were combined to carefully analyse the structural features and chemical composition of MoS_2 sheets [2]. The obtained results showed that impurity elements from the precursor were incorporated into the nanosheets during synthesis, pointing towards the necessity to carefully control the synthesis environment to avoid contamination. These findings also suggest a strategy to optimize materials performance, which we now exploit for targeted materials design (see p. 198). One example is the intentional synthesis of Pd nanoparticles with different levels of B doping [3]. Analysis by STEM and APT, complemented by density functional theory calculations and cyclic voltammogram measurements, enable us to assess changes in the catalytic activity of the nanoparticles towards the hydrogen evolution and/or hydrogen oxidation reactions (HER, HOR) due to the presence of impurities, and their effect on the grain boundary cohesion and hence, material service lifetime.

Another example focuses on the role of Sn in enhancing the efficiency of hematite (α-Fe_2O_3) for water splitting [4]. Looking both at advantages and limitation of Sn-doping of hematite thin films photoanodes, the study reveals that Sn ions at the surface eliminate surface states, preventing hole trapping and thus, enhancing the catalytic activity towards the oxygen evolution reaction (OER) on the (0001) hematite surface. An aberration corrected STEM image, the corresponding elemental distribution obtained by electron energy loss spectroscopy (EELS) and APT of a sample where Sn is located only within the top few nanometre of the hematite film is given in Fig. 1. On the other hand, the homogeneous distribution of Sn throughout the thickness of a hematite film impairs crystallization, due to grain boundary segregation of Sn, leading to inferior performance.

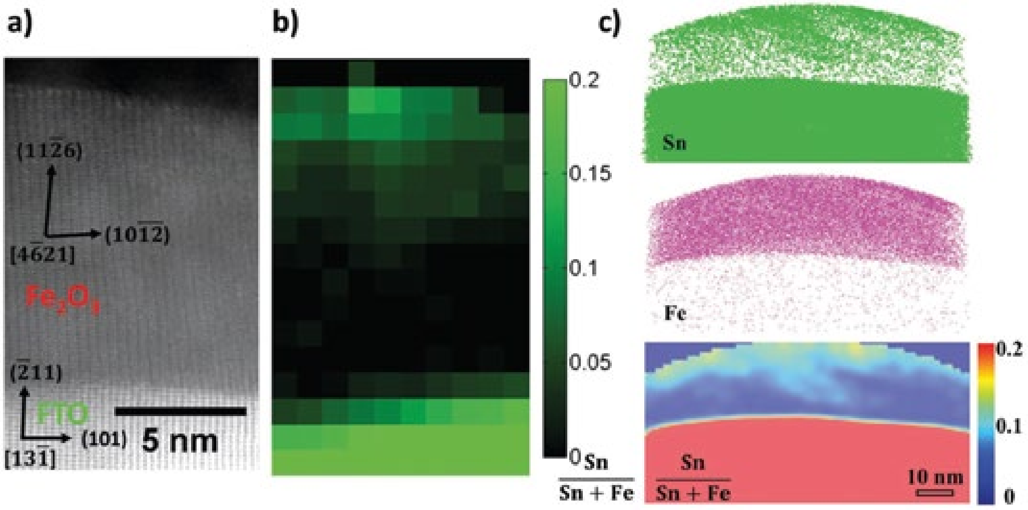

Fig. 1: *a) Atomic column resolved STEM image of a thin hematite film where Sn dopants are located only at the top surface and few nanometres below. b) EELS elemental map resembling the distribution of the Sn. c) APT reconstruction of the same sample showing the Sn and Fe distribution in 3D. Figure taken from [4].*

Next to high chemical activity towards a targeted catalytic reaction, the development of a sustainable energy economy requires longevity of the used catalytic materials. The latter is a particular obstacle, given the harsh electrochemical environments developing during reactions. Oxides show an enhanced stability in such conditions and may therefore, provide blue-prints for achieving long-term stability of catalytic materials. With this in mind, several of our studies focused on different aspects of the oxides using Ir and Ru, aiming to understand the connection between structure, stability and catalytic activity. These studies reveal the role of lattice oxygen atoms in rutile IrO_2 with respect to stability, surface hydrogenation and amorphization, and catalytic activity through a direct visualisation of the electrochemically active body of the catalyst [5, 6]. They also show that the local microstructure controls the growth rate of individual oxide grains and how crystalline defects act as diffusion pathways during oxidation in different $Ir_xRu_yO_2$ [7]. Other studies specifically target the effect different electrolytes have on the degradation, as exemplified by the example of the dissolution behaviour of the $BiVO_4$ photoanode [8]. Ongoing studies deal with $Fe_xMn_{1-x}O_2$ as electrocatalyst. With the help of Raman spectroscopy and TEM, the effect of the crystal structure as well as the morphology (nanosheets, nanocones and nanotubes) on the (photo) electrochemical performance are explored. Focus is placed on the stability of the nanostructures upon electrochemical cycling, which we will explore by identical location TEM experiments (IL-TEM).

References

1. Rurainsky, C.; Garzón Manjón, A.; Hiege, F; Chen, Y.-T.; Scheu, C.; Tschulik, K.: J. Mater. Chem. A 8 (2020) 19405.
2. Kim, S.-H.; Lim, J.; R. Sahu, Kasian, O.; Stephenson, L.T.; Scheu, C.; Gault, B.: Adv. Mater. 32 (2020) 1907235.
3. Kim, S.-H.; Yoo, S.-Y.; El-Zoka, A.A.; Lim, J.; Jeong, J.; Schweinar, K.; Kasian, O.; Stephenson, L.T.; Scheu, C.; Neugebauer, J.; Todorova, M.; Gault, B.: in preparation.
4. Hufnagel, A.G.; Hajiyani, H.; Zhang, S.; Li, T.; Kasian, O.; Gault, B.; Breitbach, B.; Bein, T.; Fattakhova-Rohlfing, D.; Scheu, C.; Pentcheva, R.: Adv. Funct. Mater. 28 (2018) 1804472.
5. Schweinar, K.; Gault, B.; Mouton, I.; Kasian, O.: J. Phys. Chem. Lett. 11 (2020), 5008.
6. Kasian, O.; Geiger, S.; Li, T.; Grote, J.-P.; Schweinar, K.; Zhang, S.; Scheu, C.; Raabe, D.; Cherevko, S.; Gault, B.; Mayrhofer, K.J.J.: Energy Environ. Sci. 12 (2019) 3548.
7. Schweinar, K.; Nicholls, R.L.; Rajamathi, C.R.; Zeller, P.; Amati, M.; Gregoratti, L.; Raabe, D.; Greiner, M.; Gault, B.; Kasian, O.: J. Mater. Chem. A. 8 (2020) 388.
8. Zhang, S.; Ahmet, I.; Kim, S.-H.; Kasian, O.; Mingers, A.M.; Schnell, P.; Kölbach, M.; Lim, J.; Fischer, A.; Mayrhofer, K.J.J., Cherevko, S.; Gault, B.; van de Krol, R.; Scheu, C.: ACS Appl. Energy Mater 3 (2020), 9523.

Fe–Al - Sustainable alloys for demanding applications

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Iron aluminide (Fe–Al) alloys are materials based on the intermetallic phases Fe_3Al and FeAl [1, 2]. Due to their relatively high Al content they form Al_2O_3 scales in oxidising environments. Specifically α-Al_2O_3 scales, which are dense, thin and adherent, are very protective against degradation of Fe–Al in aggressive environments [3, 4]. Also due to their Al content, a passive hydroxide layer forms in humid air, which acts as a lubricant, thereby minimising friction and contributing to the excellent wear behaviour of these alloys.

The high Al content of more than 20 at.% also makes the alloys a light-weight material. The density of Fe-Al alloys is about 5.7–6.7 g/cm^3, i.e. only about 80 % of that of Cr-Ni-steels or about 70 % of Ni-base superalloys. As these are typical candidates that can be possibly replaced by Fe-Al, their substitution has a number of advantages [1, 2]

- For moving parts, e.g. turbine blades, fuel saving can be expected due to the lower weight of Fe–Al.
- Better corrosion and wear resistance of Fe–Al should result in less maintenance and longer lifetime.
- The excellent corrosion resistance may also enable higher operation temperatures, thereby further reducing energy consumption.
- For large industrial facilities, support structures could be much leaner, e.g. if for a steam cracker Fe–Al tubes would be employed.

However, for industries the comparative low price of Fe–Al is of foremost interest. Fe and Al are non-strategic elements, which are actually the two most abundant metals in the earth's crust, and Fe-Al needs comparatively little alloying for fine-tuning the properties. Consequently, there has been a large number of industrial projects within recent years, investigating various processing routes for Fe–Al and for a variety of applications [5]. It has been shown that Fe–Al can be processed on standard equipment, e.g. by induction melting in air, by sand, permanent mould or investment casting, by forging, or even by additive manufacturing [6].

"Process development for economic and efficient turbine parts made of iron aluminides (Fe–Al)" is one of these ongoing MPIE projects with partners from academia and companies of different industry branches. It builds up on previous projects, where Fe–Al–Ta alloys had already been qualified for such applications. The part where the MPIE is involved in the project now aims to elucidate whether Fe–Al–Nb could replace this more costly alloy [7].

Insufficient strength above 500 °C has for long time been an obstacle for a wider application of Fe–Al alloys [1, 2]. Specifically, at the MPIE a number of alloying strategies have been developed, which yielded Fe–Al alloys whose creep resistance matches or even surpasses that of advanced steels and of plain Ni-base alloys at 700 °C. One of these strategies relies on the formation of a metastable Heusler phase. The phase could either act as a strengthening phase by forming a fine-scaled, coherent microstructure with the matrix or through aimed transformation into the stable Laves phase. The basic mechanism of the latter transformation and how it can be influenced by doping, mechanical deformation or heat treatments are understood [8]. However, for a quantitative analysis of the transformation, specifically the chemical composition of the Heusler phase, but also their morphology and distribution within the matrix, have to be quantified. As the metastable precipitates are nanometre-sized, atom probe tomography (APT) is very well suited for their analysis.

APT provides three-dimensional chemical information at the sub-nanometre resolution [9], which allows us to study the formation of these fine $L2_1$ precipitates and their compositional evolution during heat treatments. Site specific samples can be extracted using a focused ion beam (FIB) system to pinpoint exact areas and phases of interest and compare different alloy systems [10].

Fig. 1 compares reconstructions of the $L2_1/DO_3$ microstructure of Fe–25.9Al–1.6Ta (in at.%) to Fe–24.4Al–2Nb–0.03B, showing unprecedented details of the fine rectangular arrays of precipitates that formed during annealing at 700 °C. Though it is known that Heusler phases of the type Fe_2AlX may have extended homogeneity ranges to leaner compositions of X than the stoichiometric 25 at.% X, the current results are quite surprising. When comparing the composition of these small precipitates, it becomes apparent that although the Al content is similar in the Heusler phase in both alloys and close to the stoichiometric value, the precipitates in the Fe–Al–Ta alloy are with about 19 at.% Ta closer to the stoichiometric composition compared to the Nb-lean precipitates, which only contain about 10 at.% Nb.

A time-temperature-transformation (TTT) diagram established from literature data and present work suggests that at a given temperature the metastable Heusler phase transforms more readily to the stable Laves phase in the Fe–Al–Nb system [7]. Before the APT investigations had been performed, it was inferred that the difference is due to the faster diffusion of Nb. The Heusler phase cannot transform directly into the Laves phase, but dissolves first in the Fe–Al matrix before nuclei of the Laves phase preferentially form on the grain boundaries [8].

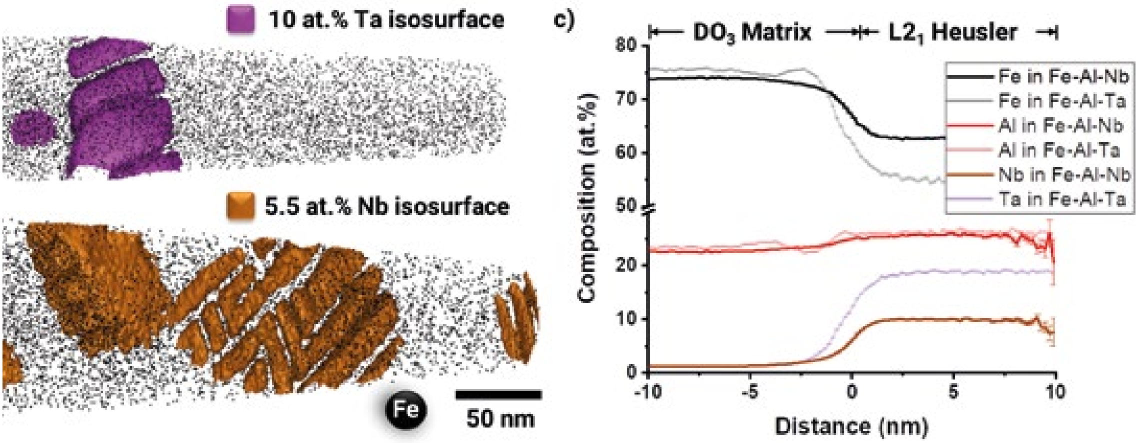

Fig. 1: *3D reconstructions of the a) Fe–Al–Ta alloy and b) Fe–Al–Nb alloy delineating precipitates with the respective isoconcentration surfaces, and c) compositional profiles across the precipitate/matrix interfaces for both alloys.*

The APT measurements now reveal that the difference between the Nb and Ta contents in the Heusler phase and the matrix is much larger in case of the Ta alloy, because the content of Nb or Ta in the Fe–Al matrix is 1.1 at.% in both alloys. Therefore, in addition to the slower diffusion kinetics it should also take more time for equilibrating the Ta-rich Heusler phase. Moreover, the precipitates in the Fe–Al–Ta alloy look more compact than the feathery appearance in the Fe–Al–Nb alloy (Fig. 1a, b). The latter results in a much larger surface area with the matrix, which could also contribute to a quicker dissolution. However, the current investigation gives no indication about coherency stresses between the precipitates and the matrix, which also have a stabilising effect for the metastable precipitates.

Further work will be carried out to gain insight on how the compositions of the Heusler phase vary with aging time and temperature, and what sort of compositional modulations contribute to the refinement of the subsequent Laves phase population. Knowledge on precipitate morphology and composition evolution will allow us to fine-tune the alloy chemistries and processing to achieve superior properties.

References

1. Palm, M.; Stein, F.; Dehm; G.: Annu. Rev. Mater. Res. 49 (2019) 297.
2. Palm, M.; Stein, F: Iron-Based Intermetallics, in Rana, R. (ed.) High-Performance Ferrous Alloys, Springer Nature Switzerland AG, Cham, Switzerland (2021) 423.
3. Peng, J.; Moszner, F.; Rechmann, J.; Vogel, D.; Palm, M.; Rohwerder, M.: Corros. Sci., 149 (2019) 123.
4. Zavašnik, J.; Peng, J.; Palm, M.; Corros. Sci.: 179 (2021) 109170.
5. Moszner, F.; Peng, J.; Suutala, J.; Jasnau, U.; Damani, M.; Palm, M.: Metals, 2019 (2019) 847.
6. Michalcová, A.; Senčekova, L.; Rolink, G.; Weisheit, A.; Pešička, J.; Stobik, M.; Palm, M.: Mater. Design 116 (2017) 481.
7. Gedsun, A.; Stein, F.; Palm, M.: MRS Adv. 6 (2021) 176.
8. Prokopčáková, P.; Švec, M.; Palm, M.: Int. J. Mater. Res.107 (2016) 396.
9. Antonov, S.; Isheim, D.; Seidman, D.; Sun, E.; Helmink, R.; Tin, S.: Superalloys 2016, 13 (2016) 199.
10. Antonov, S.; Isheim, D.; Seidman, D.; Tin, S.: Materialia 7 (2019) 100423.

Improving the performance of thermoelectric materials by microstructure design

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Ecological concerns drive the exploration of “green” alternatives to generate electricity. One environmentally-friendly approach is to recycle waste heat by thermoelectric devices to realize the heat-to-electricity conversion. These devices are without moving parts, noise- and carbon-emission free, and can be miniaturized and combined with other energy conversion technologies such as direct solar thermal energy conversion. Their conversion efficiency is defined by a dimensionless figure of merit *ZT*, which relates the electrical conductivity σ, the Seebeck coefficient *S*, the temperature *T,* and the thermal conductivity κ as follows: $ZT = \frac{S^2\sigma\cdot T}{\kappa}$. Substantial research & industrial efforts are pursued to optimize the thermoelectric performance by doping and isoelectronic alloying, i.e. tuning the alloy’s composition. However, the role of microstructure, and the interaction of dopants with lattice imperfections, are rarely considered and studied, which limits the opportunity for science-driven optimization of the material.

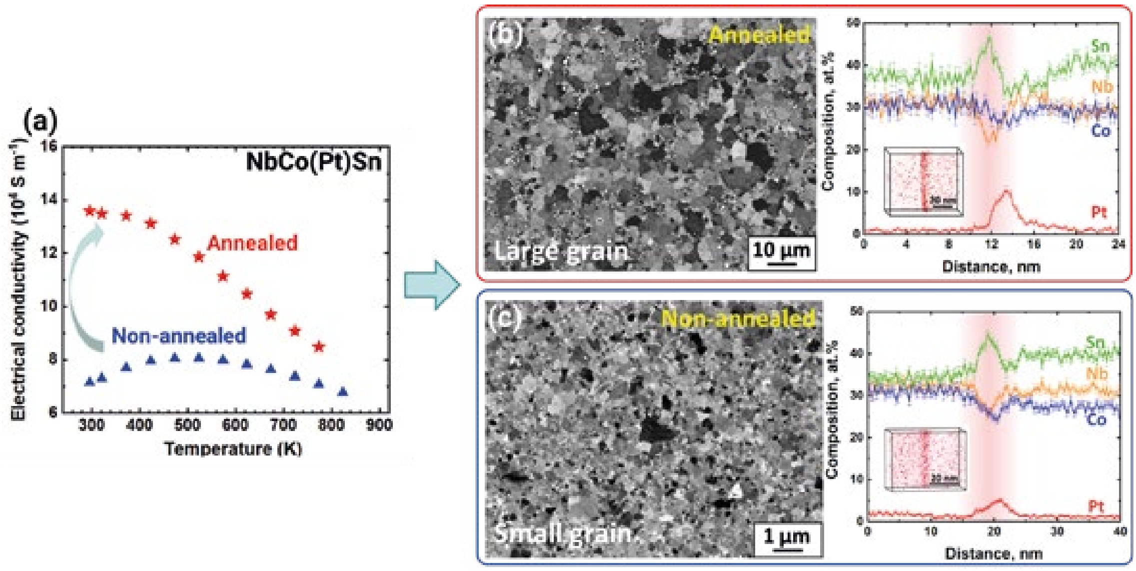

Fig.1: *Electrical conductivity of NbCo(Pt)Sn as a function of temperature: annealed vs. non-annealed sample. Grain size and grain boundary segregation for the (b) annealed and (c) non-annealed sample*

Our interdepartmental activities aim to address this knowledge gap by characterizing the multiscale complexity of the microstructure of a range of thermoelectric materials including half- and full-Heuslers [1, 2], $AgSbTe_2$ [3], and PbTe [4] and correlating it with their thermal and electrical conductivity. This will ultimately enable us to develop thermoelectric materials by microstructure design.

We will showcase our approach here with results on Half-Heusler (HH) compounds, with a stoichiometry NbCoSn, that stand out as state-of-the-art thermoelectric materials for medium-to-high temperature applications. We studied Pt-doped NbCoSn as part of a collaboration with C. Felser (Max Planck Institute for Chemical Physics of Solids). Pt substitutes Co, thereby providing an additional electron, making it an effective dopant. Pt-doping leads to significant improvements in the electrical power factor and reduces lattice thermal conductivity, as reported for $NbCo_{1-x}Pt_xSn$ (x=0.00-0.15) [5]. Interestingly in this series, the sample with x=0.05 (annealed, NbCoSn-Pt-AN) exhibits a striking increase in electrical conductivity compared to x=0.06 (non-annealed, NbCoSn-Pt), as shown in Fig. 1(a).

Grain size. The stored deformation energy induced by ball-milling drives grain growth during the post-sintering annealing. It leads to a tenfold increase in grain size (0.23 ± 0.12 µm to 2.38 ± 1.65 µm) and thus a largely reduced grain boundary (GB) area of NbCoSn-Pt-AN, as revealed

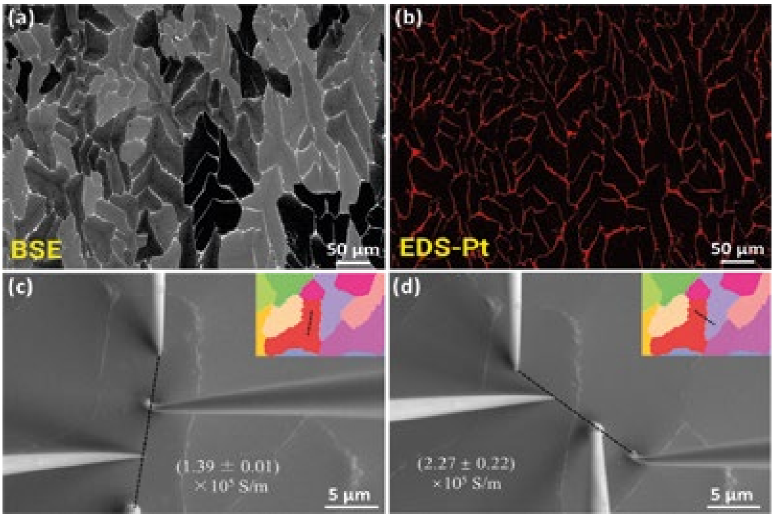

Fig. 2: *(a) Backscattered electron (BSE) image of the NbCo(Pt)Sn sample before ball milling. (b) The corresponding energy-dispersive X-ray spectroscopy (EDX) map of Pt. The white colour in the BSE image is due to the enrichment of Pt at grain boundaries. The position layout of the four needles used for the local measurement (c) within the grain interior, and (d) crossing a high angle grain boundary, which was assessed by electron-backscattered diffraction (inset).*

by backscattered electron imaging in a scanning electron microscope (SEM) (Fig. 1b and c). The difference in grain size between the two samples becomes the first key microstructural factor influencing the electrical conductivity through grain boundary scattering.

Grain boundary segregation. Atom probe tomography shows that the intragranular Pt-content is similar for both alloys, i.e. ~1.35 ± 0.09 at.% for NbCoSn-Pt and 1.27 ± 0.09 at.% for NbCoSn-Pt-AN. The improvement in electrical conductivity does hence not arise from the doping level, rather, from the microstructure evolution. Pt-segregation to GBs is found in both samples Fig.1 (b) and (c), with varying degree of Pt enrichment across different GBs, likely depending on their geometric characteristics.

Local electrical conductivity. To assess the impact of this GBs segregation of Pt on the electrical conductivity, we used an *in situ* four-point-probe technique within an SEM to measure the local electrical conductivity [6]. As the grain size must be sufficiently large to separate the four probes, we selected the NbCoSn-Pt (x=0.05) sample before ball milling with an average grain size of approx. 25 µm. The corresponding backscattered electron micrograph and compositional map from energy-dispersive X-ray spectroscopy for Pt are respectively shown in Fig. 2 (a) and (b), highlighting Pt enrichment and percolation along grain boundaries. The electrical conductivity was measured inside two adjacent grains and across the corresponding high angle grain boundary, as assessed by electron-backscattered diffraction (inset in Fig. 2c and d). One example of the position layout of the four probes is displayed in Fig. 2(c) for grain interior and Fig. 2(d) for a high angle grain boundary. The absolute value of the electrical conductivity is (1.39 ± 0.01) $\times 10^5$ S/m for the grain interior, i.e. comparable with the value of NbCoSn-Pt-AN at room temperature. Across the GB, and despite possible space-charge effects, the conductivity increases almost twofold to (2.27 ± 0.22) $\times 10^5$ S/m thanks to the enrichment of Pt at GBs. We therefore prove the beneficial effect of GB-segregation on the electrical conductivity. It is hence possible to use grain boundary engineering to manipulate the GB-transport properties, opening new possibilities for optimizing TE performance.

Generalization. The philosophy and approach that was discussed here was deployed to analyze several other materials, for instance, PbTe and AgSbTe2 (see p. 13), Ti(Co, Fe)Sb Half-Heusler with the collaboration with G. J. Snyder (Northwestern University, USA), and the Fe2AlV full-Heusler alloy that is being synthesized in-house. For the Fe2AlV, we also investigated the possibilities of using the selective-laser melting machine to manipulate the microstructure by surface-laser remelting [7], which can be seen as a precursor to additive manufacturing. We demonstrated an influence of grain boundary and structural defects on both electrical and thermal conductivities measured locally. We are also exploring the overlooked effect of impurity introduction during processing for instance.

References

1. Gomell, L.; Roscher, M.; Bishara, H.; Jägle, E.A.; Scheu, C.; Gault, B.: Scr. Mater. 193 (2021) 153.
2. Luo, T.; Serrano-Sánchez, F.; Bishara, H.; Zhang, S.; Bueno Villoro, R.; Kuo, J.J.; Felser, C.; Scheu, C.; Snyder, G.J.; Best, J.P.; Dehm, G.; Yu, Y.; Raabe, D.; Fu, C.; Gault, B.: Acta Mater. 217 (2021) 117147.
3. Abdellaoui, L.; Zhang, S.; Zaefferer, S.; Bueno-Villoro, R.; Baranovskiy, A.; Cojocaru-Mirédin, O.; Yu, Y.; Amouyal, Y.; Raabe, D.; Snyder, G.J.; Scheu, C.: Acta Mater. 178 (2019) 135.
4. Abdellaoui, L.; Chen, Z.; Yu, Y.; Luo, T.; Hanus, R.; Schwarz, T.; Bueno Villoro, R.; Cojocaru-Mirédin, O.; Snyder, G.J.; Raabe, D.; Pei, Y.; Scheu, C.; Zhang, S.: Adv. Funct. Mater. (2021) 2101214.
5. Serrano-Sánchez, F.; Luo, T.; Yu, J.; Xie, W.; Le, C.; Auffermann, G.; Weidenkaff, A.; Zhu, T.; Zhao, X.B.; Alonso, J.A.; Gault, B.; Felser, C.; Fu, C.: J. Mater. Chem. A 6 (2020) 4883.
6. Bishara, H.; Ghidelli, M.; Dehm, G.: ACS Appl. Electron. Mater. 2(7) (2020) 2049.
7. Gomell, L.; Haeger, T.; Roscher, M.; Bishara, H.; Heiderhoff, R.; Riedl, T.; Scheu, C.; Gault, B.: arXiv:2107.07327. (2021).

III-V Semiconductors and alloys at the nanoscale: materials design for novel optoelectronic and elastic properties

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Designing and controlling the nanoscale structure of semiconductors and alloys is a promising strategy in order to create materials with targeted optoelectronic and mechanical properties. In this context, III-V and III-N nanostructures are particularly attractive candidates for surmounting materials-related challenges in applications ranging from optoelectronics, power electronics and hydrogen diffusion barriers to hard and wear resistant coatings.

One possible route to create such nanostructured materials is to use colloidal nano-scale building blocks, e.g., nanocrystals, as "*artificial atoms*" and assemble them into **quantum dot (QD) solids** (Fig. 1). III-V semiconductor-based QDs, consisting of e.g., InP or InGaP nanocrystals, are among the most widely investigated materials systems due to their potentially superior optical properties and lower toxicity, compared to commercially available II-VI QD solids. Nevertheless, III-V systems still suffer from inferior colour purity due to broad emissivity and poor photoluminescence quantum yields. We recently demonstrated [1] that nanocrystal stoichiometry dispersion is a major source of trap states and largely responsible for the observed emission broadening in III-V QD solids. Whereas II-VI QD solids require only nanocrystal size control to achieve narrow emission, it is necessary to realize in addition nanocrystal stoichiometry control in the case of III-V QD solids.

Anomalously strong temperature-induced shifts of the electronic bandgaps are another important source of emission broadening. In order to reveal the origin of these anomalous shifts, we used so-called magic-sized nanoclusters as atomically precise model systems for nanocrystals with zero dispersion in both size and stoichiometry. Tantalizingly, approaches that are commonly employed to accurately predict temperature-dependent bandgaps in bulk materials have been known for more than a decade to yield qualitatively wrong results in the case of nanocrystals. We therefore developed a novel approach that allows us to accurately compute temperature-induced band gap shifts also for nanocrystals, using constrained *ab initio* molecular dynamics (AIMD) simulations on the excited state potential energy surface and atomistic thermodynamics, where the band gap is computed as the Gibbs free energy of exciton formation.
The key innovation of our approach in contrast to existing methods is to take into account the fully anharmonic exciton-phonon coupling [2].

We revealed [2] that the experimentally observed anomalous colour shifts with temperature are caused by excitonic bond-softening: in nanocrystals, the excitons are distributed over only a small number of atoms due to the strong quantum confinement. In consequence, the occupation of antibonding states due to the presence of the excitons significantly weakens the interatomic bonds, leading to a pronounced exciton-induced red shift of the phonon density of states (PDOS). This shift is almost two orders of magnitudes larger than the corresponding one observed in bulk materials. The red-shifted PDOS in turn is responsible for the anomalous bandgap shift due to its impact on the Gibbs free energy of exciton formation. Our results underline the importance of explicitly considering exciton-electron-phonon coupling in nanostructures and suggest how to design systems with improved optical properties.

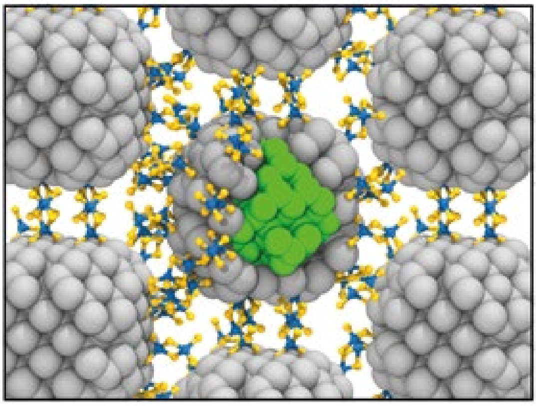

Fig. 1: *Schematic representation of a QD solid, consisting of core/shell nanocrystals assembled into a superlattice. Chalcogenidometallate complexes are used as surface-selective linkers and "conductive glue", connecting the nanocrystals both mechanically and electrically.*

Moreover, the design of **heterostructures at the nanoscale**, such as multilayered structures consisting of CrN and AlN layers with a few nm thickness allows us to tune and tailor the mechanical properties for hard coating applications. In order to establish relations between the elastic properties, chemical composition and crystal structure, we investigated fast screening methods for fracture [3], but also applied novel scanning transmission electron microscopy-electron energy loss spectroscopy (STEM-EELS) methods to measure mechanical properties with nm high spatial resolution [4]. We analyzed AlN/CrN multilayers deposited epitaxially on MgO together with colleagues from Montanuniversität Leoben and TU Wien. The AlN layer thickness was increased from initially 1 nm to 10 nm, while the CrN layer thickness was kept

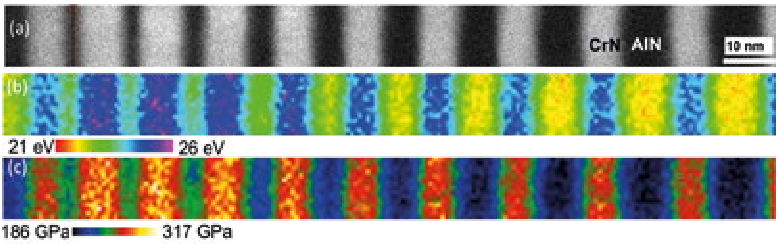

Fig 2: *a) STEM-HAADF (high-angle annular dark field) image of CrN (bright) and AlN (dark) layers grown on MgO, with AlN layer thickness increasing from 1 nm to 10 nm from left to right. (b) Corresponding low loss EELS plasmon maps and (c) bulk modulus based on an empirical relation between plasmon energy and bulk modulus. Taken from [4].*

constant at 4 nm (Fig. 2). Atomically resolved S/TEM reveals that AlN layers up to 4 nm grow in the metastable zincblende structure, while thicker AlN layers possess the room temperature stable wurtzite structure. The difference in crystal structure is also seen in the fine structure of the N-K edge in core loss EEL spectra as well as a shift in the plasmon peak positions for low loss EELS. The plasmon energy and the bulk modulus, which are related by an empirical relation established in literature were quantitatively extracted. Metastable cubic AlN, which allows for improved fracture toughness, was revealed to feature the highest bulk modulus values with 233±9 GPa in agreement with density functional theory (DFT) predictions [4].

Epitaxial strain is the origin of the long standing and highly debated issue of threading dislocations in III-Nitride materials. Nevertheless, the topic has recently been reinvigorated due to the detrimental effect they have on power electronic devices. An intriguing feature of these defects is that experimental evidence suggests that screw dislocations are associated with open core structures, thus forming **nanopipes (NPs)**. Although NPs exhibit large free surface areas which increase the energy, they can be energetically favourable if the surface energy is compensated by the core and strain energy of the material removed to create the NP.

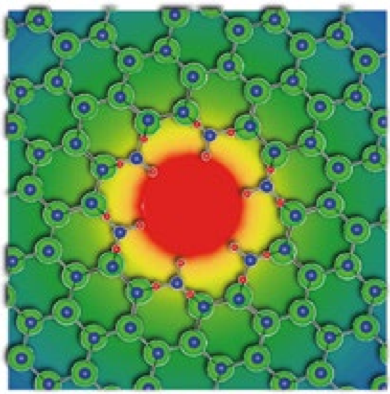

Fig. 3: *Schematic representation of the energy density associated with a full core c-type screw dislocation in GaN superimposed with the atomic structure of a hollow core defect. Hot/cold colours indicate higher/lower energy values, respectively. Green, blue and red balls indicate Ga, N, and H atoms, respectively.*

In a recent study we combined DFT calculations with elasticity theory and we investigated the mechanisms governing the formation and the properties of nanopipes. Based on these calculations we constructed a screw dislocations' phase diagram, which describes the energetically most favourable core structures as function of the species' chemical potentials. A general trend that emerges from this diagram is that nanopipes are energetically favourable under typical MOCVD or MOVPE growth conditions. Under these conditions, the cation and anion dangling bonds at the open core inner surfaces are passivated by NH_x molecules and H atoms, respectively (Fig. 3). This reduces considerably the surface energy and the formation of nanopipes with hexagonal cross section and equilibrium diameters ranging for ≈1 to ≈2 nm is energetically favourable. Nanopipes offer routes for efficient diffusion and the inner free surface sites for preferential incorporation of impurities. Indeed, our calculations indicate that both C and Mg dopants decorate the inner surface at concentrations (in at. %) at least one order of magnitude higher than in highly doped bulk semi-insulating layers [5].

The aforementioned examples from the fields of opto- and power electronics and hard coatings demonstrate that with our new methodological advances, both computational and experimental, it is now possible to reveal the complex interplay between the properties of materials at the nanoscale and their interaction with a complex growth environment. They also illustrate how our continued methodological developments guide novel applications design, and constitute an essential component in cutting-edge materials research and technology.

References

1. Rusishvili, M.; Wippermann, S.; Talapin, D.; Galli, G.: Chem. Mater. 32 (2020) 9798.
2. Muckel, F.; Lorenz, S.; Yang, J.; Nugraha, T. A.; Scalise, E.; Hyeon, T.; Wippermann, S.; Bacher, G.: Nature Comm. 11 (2020) 4127.
3. Völker, B.; Du, C.; Fager, H.; Rueß, H.; Soler, R.; Kirchlechner, C.; Dehm, G.; Schneider, J. M.: Surf. Coat. Technol. 390 (2020) 125645.
4. Zhang, Z.; Chen, Z.; Holec, D.; Liebscher, C. H.; Koutná, N.; Bartosik, M.; Zheng, Y.; Dehm, G.; Mayrhofer, P. H.: Acta Mater. 194 (2020) 343.
5. Koller, C.; Lymperakis, L.; Pogany, D.; Pobegen, G.; Ostermaier, C.: J. Appl. Phys. 130 (2021) 185702.
6. Yoo, S-H.; Lymperakis L.; Neugebauer J.: Phys. Rev. Mater. 5 (2021) 044605.

Defect phases –
Thermodynamics and influence on material properties

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To design novel alloys with tailored properties and microstructure, two materials science approaches have proven immensely successful: Firstly, thermodynamic and kinetic descriptions for tailoring and processing alloys to achieve a desired microstructure. Secondly, crystal defect manipulation to control strength, formability and corrosion resistance. However, to date, the two concepts remain essentially decoupled. A bridge between them is needed to achieve a single conceptual framework. A powerful concept to establish such a bridge are defect phase diagrams, which provide systematic tools to describe and analyse the possible chemical and structural states that specific defects can have as function of key state variables, such as chemical composition, temperature or strain.

Scientists from the RWTH Aachen University and MPIE established a large scale collaborative research centre (CRC) funded by the German Research Foundation (DFG) (see p. 76) aiming at a fundamentally new approach to achieve this goal. The key concept are defect phase diagrams, which describe the coexistence of and transitions between defect phases. The availability of such a framework will enable a paradigm shift in the description and design of future engineering materials [1].

Formally, a 'defect phase' is a structurally and chemically distinct atomic-scale defect configuration for which all physical properties can be expressed as smooth (continuous and infinitely differentiable) functions of intensive control variables such as temperature or chemical potential. A structure can only be considered a defect phase, if it is spatially confined at or next to the defect.

While the concept of defect phases has been theoretically postulated and discussed more than four decades ago, their actual observation became only possible recently with the availability of experimental techniques with atomic resolution such as aberration corrected transmission electron microscopy (TEM) and atom probe tomography (APT). For example, in a correlative study of both techniques, a local change of structure and chemistry next to dislocations in high-Mn austenitic steel and in Au-Pt alloys has been observed in the MA department [2, 3]. Nevertheless, using these highly advanced experimental techniques to systematically map defect phases (left side of Fig. 1) would be too time-consuming and due to a sensitive dependence on the thermodynamic state variables rather challenging. In the CRC we therefore aim at an alternative strategy to identify defect phases, namely guided probing. Here, physical properties of materials such as the elastic modulus or the corrosion behaviour are monitored in order to identify abrupt changes as function of the chemical potential of the involved species (right side of Fig. 1).

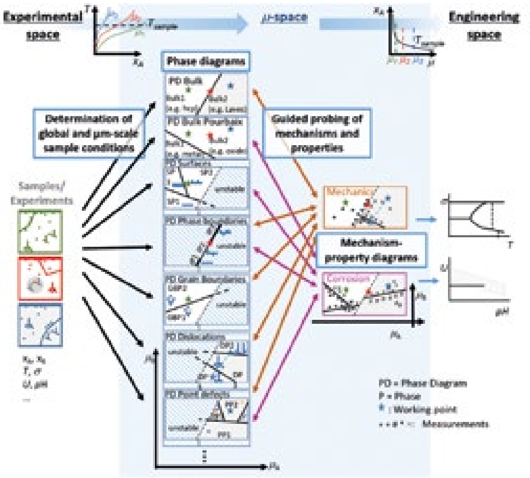

Fig. 1: *Modelling framework of defect phase diagrams. The experimental space visualizes the experimental degrees of freedom. The variety of phase diagrams for bulk and all kinds of defects is represented in the μ-space. The engineering space considers parameter dependent materials properties. Adopted from Ref. [1].*

Since the chemical composition at and around defects is intrinsically inhomogeneous, e.g., due to segregation, the natural state variable is not the concentration like for bulk phases, but the chemical potential. The constancy of the chemical potential throughout the sample in chemical equilibrium (μ-space in Fig. 1) allows one to plot the phase diagram of the bulk material and of the individual defects in the same coordinate system. This property provides a direct route to connect bulk properties with those of various defects that give rise to the complex microstructure and surfaces of materials. For the latter, the MPIE has a long-standing expertise in deriving surface phase diagrams, which is now being applied and extended within the framework of the CRC to study, e.g., the impact of alloying Mg with Ca and Al on surface structure and stoichiometry (Fig. 2). Similar defect phase diagrams can also be constructed for phase boundaries, grain boundaries, dislocations, or point defects.

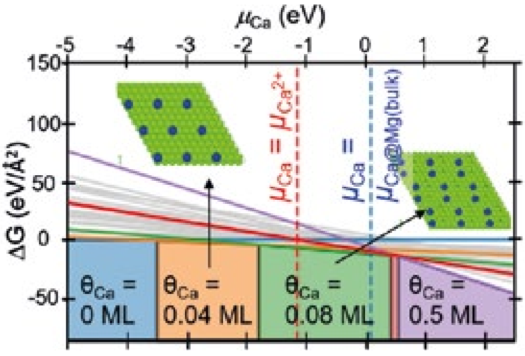

Fig. 2: *Calculated surface phase diagrams for Mg alloyed with Ca (top) and Al (bottom) in an electrochemical setup. The diagram shows as function of the Ca(Al) chemical potential the stable Ca(Al) rich phases and their compositions at Mg(0001)/water interfaces. Insets show top views of some of these structures. Dashed lines mark chemical potential conditions realized in selected measurements.*

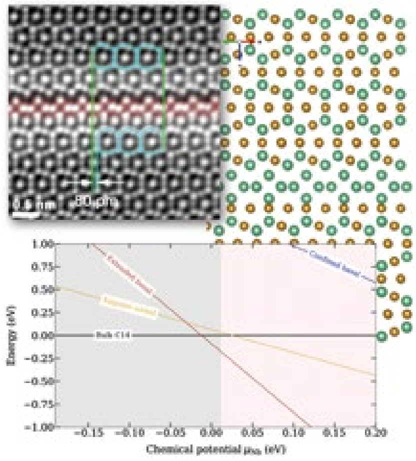

Fig. 3: *Stacking faults phases in the Nb-rich C14 Laves phase $NbFe_2$. Upper left: STEM image of an extended basal stacking fault. Upper right: Atomistic structure of the same defect as used for DFT calculations. Below: Defect phase diagram of several stacking faults (lines) as compared to the bulk phase diagram (shading). Adopted from Ref. [6].*

The rapid improvement of atomistic simulations revealed that defect phases are not rare exceptions, but should be a common feature in materials. For example, simulations in the CM department showed already in 2015 that dependent on the chemical potential of H in fcc Ni, the formation of a local hydride can be observed next to edge dislocations [4]. This is remarkable, because the hydride phase is not stable in the bulk at ambient conditions. Further extensive simulations have been performed in the CM department on grain boundaries, confirming the observation of defect phases in Al alloys by the SN department [5].

Recently, we have investigated stacking faults in the Nb-rich C14 Laves phase $NbFe_2$. The bulk phase diagram of Fe-Nb suggests a rather narrow solubility of Nb in this phase, due to the formation of the competing Nb_6Fe_7 μ-phase. Instead of this phase, however, a large number of various types of planar defects is observed in experiment [6]. The atomic configurations of these defects have been characterized by aberration-corrected scanning TEM in the SN department and density functional theory (DFT) calculations in the CM department (Fig. 3). These studies reveal that some of the stacking faults contain one layer of the characteristic Zr_4Al_3 type unit of the μ-phase (red shaded crosses in the TEM image), i.e., correspond to a defect phase that is stabilized by an excess Nb content. Nevertheless, the confinement and the stacking of atomic layers does not correspond to a bulk phase, but rather fulfil the definition of defect phases provided above.

Apart from the basal defect shown in the upper part of Fig. 3 a variety of different extended and confined stacking faults, that are lying parallel to the basal or pyramidal habit planes are observed. The DFT calculations have been used to determine the relative stability of these defect structures. In the lower part of Fig. 3 the formation energy of selected stacking faults is plotted as a function of the chemical potential. The shading in grey indicates the stability region of the C14 bulk phase. Slightly below the phase equilibrium of the μ-phase (vertical line at $\mu_{Nb} \approx 0.01$ eV), one can see the relative stability of the extended basal stacking observed in experiment. Other defect structures become lower in energy than the extended basal structure for certain chemical potentials, but do not lead to stable defect structures.

Based on these early successes, we envisage that defect phase diagrams will play the same role as their bulk counterparts. They will allow the selection of defects with a desired structural and chemical configuration, which in turn are the carriers of specific defect-property relations and, hence, enable the atomistically guided design of materials with tailored mechanical and corrosion properties.

References

1. Korte-Kerzel, S.; Hickel, T.; Huber, L.; Raabe, D.; Sandlöbes-Haut, S.; Todorova, M.; Neugebauer, J.: Int. Mater. Rev. (2021) DOI: 10.1080/09506608.2021.1930734.
2. Kuzmina M.; Herbig M.; Ponge D.; Sandlöbes, S.; Raabe, D.: Science 349 (2015) 1080.
3. Zhou X.; Mianroodi, J. R.; Da Silva, A. K.; Koenig, T.; Thompson, G. B.; Shanthraj, P.; Ponge, D.; Gault, B.; Svendsen, B.; Raabe, D.: Sci. Adv. 7 (2021) 1.
4. Leyson, G.; Grabowski, B.; Neugebauer, J.: Acta Mater. 89 (2015) 50.
5. Zhao, H.; Huber, L.; Lu, W.J.; Peter, N.J.; An, D.Y.; De Geuser, F.; Dehm, G.; Ponge, D.; Neugebauer, J.; Gault, B.; Raabe, D.: Phys. Rev. Lett. 124 (2020) 6.
6. Šlapákováa , M.; Zendegani, A.; Liebscher, C.H.; Hickel, T.; Neugebauer, J.; Hammerschmidt, T.; Ormeci , A.; Grin, J.; Dehm, G.; Kumar, K.S.; Stein, F.: Acta Mater. 183 (2020) 362.

Bidirectional transformation induced plasticity in dual-phase high entropy alloys

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Recently developed dual-phase high entropy alloys (HEAs) exhibit both an increase in strength and ductility upon grain refinement, overcoming the strength-ductility trade-off in conventional alloys [1]. Metastability engineering through compositional tuning in non-equimolar Fe-Mn-Co-Cr HEAs enabled the design of a dual-phase alloy composed of metastable face centered cubic (fcc) and hexagonal closed packed (hcp) phases. This microstructure emerges in a $Fe_{50}Mn_{30}Co_{10}Cr_{10}$ (at.%) alloy after cooling from the high temperature single phase region to room temperature through a partial martensitic transformation. Both fcc- and hcp-phases are single phase supersaturated solid solutions and the microstructural refinement associated with the martensitic transformation leads to an increase in ultimate tensile strength from ~650 MPa to ~850 MPa and total elongation from ~50% to ~75% when reducing the grain size from 45 µm to 4.5 µm [2].

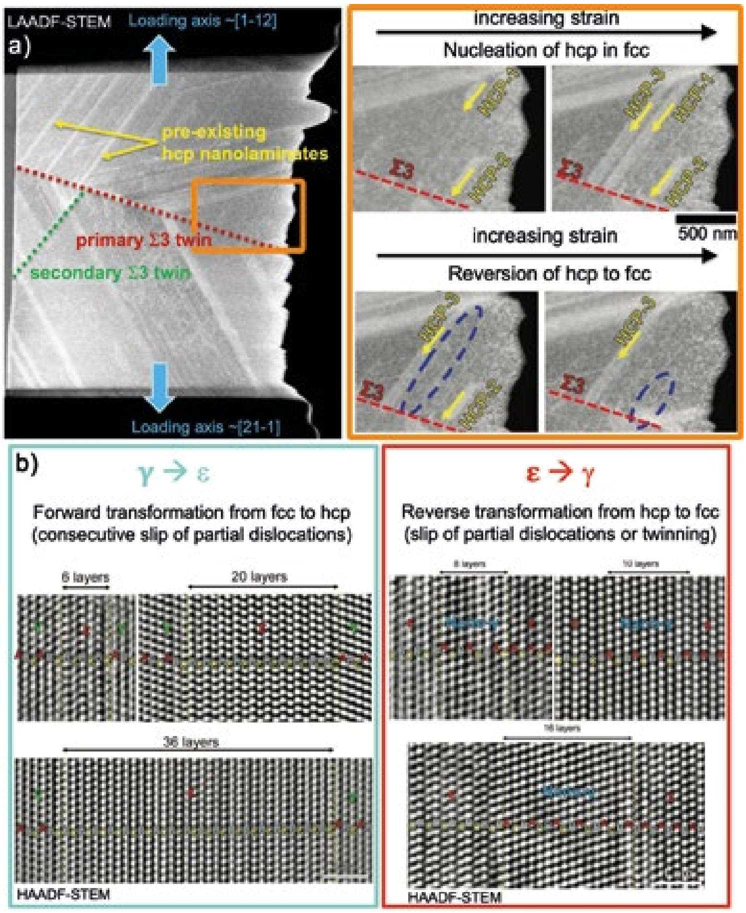

Fig. 1: *a) Low angle annular dark-field (LAADF) STEM image of the focused ion beam prepared straining sample form a fcc γ-block with pre-existing hcp ε-nanolaminates. Dynamic bidirectional forward γ→ε and backward ε→γ transformation upon straining.*
b) Atomic resolution STEM images of various configurations of ε-nanolaminates in a γ-block, respectively γ-nanotwins in a ε-block, after bulk tensile deformation.

We have used length scale bridging transmission electron microscopy (TEM) observations in combination with *in situ* straining in the scanning TEM (STEM) to unravel the underlying microstructural origin of this outstanding property combination in the dual-phase HEA ($Fe_{50}Mn_{30}Co_{10}Cr_{10}$ (at.%)). In the as-quenched condition, a hierarchical, nanolaminated structure is observed in both fcc (γ) and hcp (ε) phases. Upon bulk tensile straining, the microstructure dynamically refines and secondary strain-induced ε-nanolaminates form within the γ-block in the hcp ε-block, a high density of phase transformation induced γ-nanolaminates and -twins is developing. These static post-deformation observations suggest that both forward γ→ε and backward ε→γ transformations are the main carriers of plasticity in this metastable dual-phase high entropy alloy.

We also performed *in situ* straining in the STEM using a custom build Cu-straining support in a displacement controlled Gatan straining holder to probe the dynamic evolution of the hierarchical nanolaminated microstructure as shown in Fig. 1a. The pre-existing hcp ε-nanolaminates in the γ-block are observed to grow by the motion of their leading partial dislocation and their number density increases upon straining. Interestingly, we could observe the dynamic strain induced forward γ→ε and backward ε→γ transformation *in situ* in the region indicated by an orange rectangle in Fig. 1a, referred to as “bidirectional transformation induced plasticity” (Bi-TRIP) effect.

In the early stages of straining, the nucleation of ε-plates (HCP-1-3) is observed (Fig. 1a), which continue to grow with increasing strain until their extension is blocked by the primary S3 twin boundary. By further straining, the HCP-1 and HCP-2 plates are reverted to fcc, as well as the HCP-3 plate at a later stage (not shown here). The trailing partial dislocation unzips the hcp nanolaminates, thus establishing the underlying mechanism for a novel reversable TRIP effect.

The key ingredients controlling this bidirectional transformation are the stacking fault energy, local temperature and stress-strain fields. The stacking fault energy is estimated to ~6.5 mJ/m^2 for this dual-phase HEA promoting the forward $\gamma \rightarrow \varepsilon$ transformation and building the basis for the reverse transformation, since local temperature or strain fluctuations can initiate the reversion by the motion of trailing partial dislocations. The reverse $\varepsilon \rightarrow \gamma$ transformation depends on local stress or temperature fluctuations during straining, since the hcp ε-phase is more stable than γ at room temperature. We observed that due to such fluctuations the partial dislocations moved in some cases back, thus reversing the initial phase transformation.

This concept was further extended in an interstitial HEA by deformation-driven bidirectional transformation. Controlled cold-rolling and tempering promote the formation of dual-phase nanostructures, nano-grains and -twins in a $Fe_{49.5}Mn_{30}Co_{10}Cr_{10}C_{0.5}$ (at.%) alloy resulting in an ultimate tensile strength of 1.05 GPa and a total elongation of 35 % [3]. The key design element is the tuning of the materials stacking fault energy close to its thermodynamic limit to trigger forward and reverse phase transformations upon loading. By joint activation of Bi-TRIP and twinning induced plasticity (TWIP) an excellent strength-ductility synergy can be achieved in tempered specimens with preceding-rolling reduction of 34 %.

Obtaining reliable values of stacking fault energies (SFEs) and hence relative stabilities of the fcc- and hcp-phases in these compositionally complex alloys also at non-zero temperatures is to date a big challenge. We have developed a set of density functional theory-based methods to compute the concentration dependence of SFEs [4] including magnetic and temperature effects [5]. To account for chemical disorder, different techniques are used such as the coherent potential approximation or special quasi random structures. Many of these alloys reveal complex antiferromagnetic interactions and it turned out that for achieving agreement with experimental data it is crucial to go beyond the assumption of idealized paramagnetic alloys with a careful treatment of magnetism and magnetic fluctuations [6].

References

1. Li, Z.; Pradeep, K.G.; Deng, Y.; Raabe, D.; Tasan, C.C.: Nature 534 (2016) 227.
2. Lu, W.; Liebscher, C.H.; Dehm, G.; Raabe, D.; Li, Z.: Adv. Mater. 30 (2018) 1804727.
3. Su, J.; Wu, X.; Raabe, D.; Li, Z.: Acta Mater. 167 (2019) 23-39.
4. Guo, W.; Su, J.; Lu, W.; Liebscher, C.H.; Kirchlechner, C.; Ikeda, Y.; Körmann, F.; Liu, X.; Xue, Y.; Dehm, G.: Acta Mater. 185 (2020) 45.
5. Li, Z.; Körmann, F.; Grabowski, B.; Neugebauer, J.; Raabe, D.: Acta Mat. 136 (2017) 262.
6. Wu, X; Li, Z.; Rao, Z.; Ikeda, Y.; Dutta, B.; Körmann, F.; Neugebauer, J.; Raabe, D.: Phys. Rev. Materials 4 (2020) 033601.

Interplay of chemistry and faceting at grain boundaries in a high strength Al alloy

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Grain boundaries (GBs) are regions connecting adjacent crystals with different crystallographic orientations. GBs are a type of lattice imperfection, with their own structure and composition, and as such impact a material's mechanical and functional properties. Structural motifs and phases formed at chemically decorated GBs can be of a transient nature or are local thermodynamic structural-chemical equilibrium states. The boundary between two crystal grains can decompose into arrays of facets with distinct crystallographic characters. Faceting occurs to minimize the system's free energy, i.e., when the total interfacial energy of all facets is below that of the topologically shortest interface plane. Previously, the interplay of chemistry and faceting was revealed for specific GBs in well-defined bicrystals, which are realistically not encountered in engineering alloys. The majority of GBs in bulk metallic materials from conventional processing is of a more general type. Evidence for structural transitions in such boundaries in Al-alloys is scarce, and the interplay with chemistry is not well understood. Recently [1], we have revealed the subtle yet important interplay between the faceting of GBs and their chemical decoration with solutes in a high strength 7xxx Al alloy. We combined cutting-edge microscopy, microanalysis techniques, and atomistic simulations to demonstrate that facets exist in polycrystalline samples, ranging in length from a few to hundreds of nanometres at GBs. We also evidenced that the local chemistry is strongly correlated with the facet character.

In the present report, we highlight one example of the near Σ5 coincident site lattice boundaries in a coarse grained 7xxx Al alloy. A homogenized Al-6.22 %Zn-2.46 %Mg-2.13 %Cu (wt. %) was used for this study. The alloy was solution heat treated at 475 °C, followed by water quenching. The detailed process and precipitation behaviour during aging is described in [2]. The Σ5 grain boundary observed has a misorientation angle of 38° about a <001> common axis. Fig. 1(a) illustrates the Σ5 GB characterized by Z-contrast high-angle annular dark-field (HAADF) scanning transmission electron microscopy (STEM) performed along the common tilt <001> Al zone axis. The GB configuration contains two sets of alternating facets along the entire length, with each type highlighted by a coloured line. The orange segments are symmetric {120} facets, and the green segments are asymmetric {130} planes. Fig. 1(b) shows the energy dispersive spectroscopy (EDS) analysis from the faceted Σ5 GB. At this scale, we do not observe segregation at the {130} facets, while the {120} facets bear roughly 300 nm lathlike precipitates enriched with Mg and Cu. The HAADF-STEM image in Fig. 1(c) reveals that the precipitate has an orthorhombic structure, which matches the S phase (Al_2CuMg). The S phase is semi-coherent with the Al-matrix and has an orientation relationship of $<100>_s//<100>_{Al}$, and an interface of $\{001\}_S//\{120\}_{Al}$.

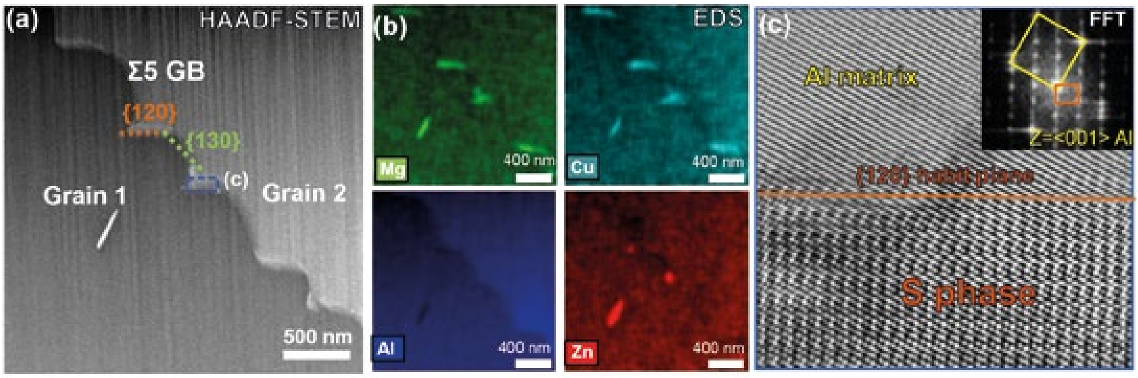

Fig. 1: *(a) HAADF-STEM image of a near Σ5 GB in the as-quenched Al-Zn-Mg-Cu alloy; (b) EDS maps of the faceted GB showing Mg and Cu enriched precipitates; (c) HAADF-STEM image of the precipitate highlighted by blue rectangle in (a), with inset of the corresponding Fast Fourier transform pattern. GB: grain boundary. Figure is from Ref. [1], published under a CC-Attribution 4.0 international license.*

We performed complementary atom probe tomography (APT) analyses on the same Σ5 GB on another location, shown in Fig. 2. Two lathlike S phases enriched in Mg and Cu are visible in Fig. 2(a), highlighted by isosurfaces delineating regions containing more than 10 at. % Mg. A composition profile across the large precipitate was calculated along a 25 nm-diameter cylinder aligned with the

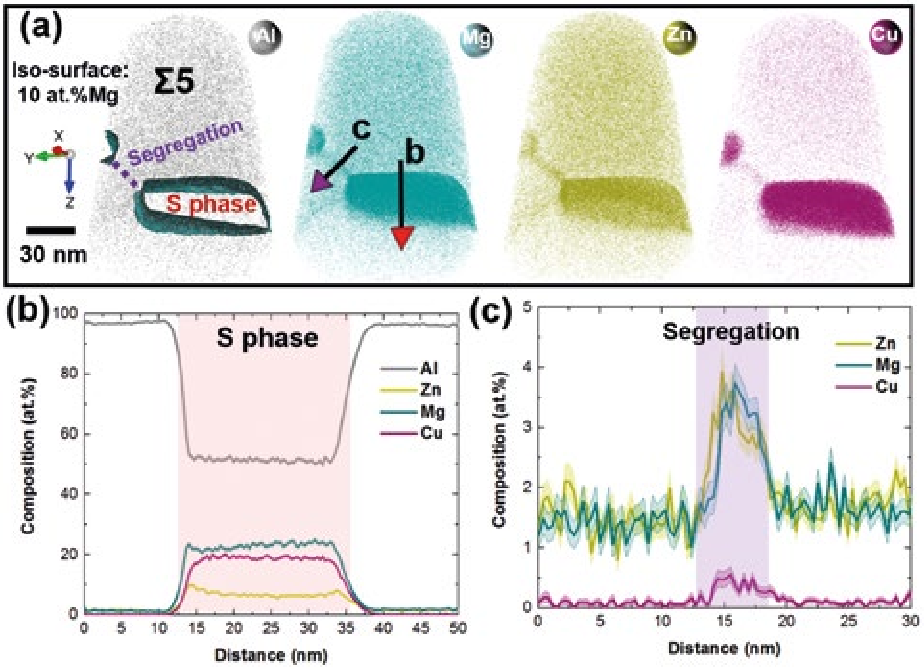

Fig. 2: *APT analysis of the same Σ5 GB with STEM analysis: (a) Atom maps of all elements; (b) Corresponding composition profile across the precipitate calculated along the red arrow; (c) Corresponding composition profile across the region between two precipitates calculated along the purple arrow. GB: grain boundary. Figure is from Ref. [1], published under a CC-Attribution 4.0 international license.*

red arrow in Fig. 2(b). It shows an average composition of 52 at. % Al, 23 at. % Mg, 18 at. % Cu, and 7 at. % Zn within the precipitate. This agrees with the stoichiometry of the S phase (Al_2CuMg). It is most likely that the 7 at. % Zn substitutes to Cu sites, as Cu and Zn are both undersized, and the combined compositions of Cu (18 at. %) and Zn (7 at. %) equals 25 at. %, as expected for the $Al_2Mg(Cu/Zn)$ stoichiometry. At this more detailed scale, we also observe Zn, Mg, and Cu enrichments along the {130} facet that connects the two precipitates and the corresponding composition profile is plotted in Fig. 2(c). The average composition in the enriched region are 3.1 at. % Zn, 3.1 at. % Mg and, 0.5 at. % Cu, which are enrichment factors of two in Zn and Mg and 3 in Cu compared to the abutting bulk solute composition. Combining the STEM and APT results, we conclude that the S phase nucleates on the {120} facets, whereas solutes segregate to the {130} facets of the faceted Σ5 GB.

We then used atomistic simulations to better understand the interplay of the chemistry and the structural faceting of the Σ5 GB. We examined the formation of a Mg-rich phase near the symmetric {120} and {130} 36.87° <001> Σ5 GBs by using the Variance Constrained Semi-Grand Canonical Molecular Dynamics/Monte Carlo (MD/MC) approach [3]. Both GBs experience various stages of segregation, from the initial decoration of the GB and adjacent planes, to the nucleation of a Mg-rich precipitate. However, the underlying energy landscape of the {120} boundary drives the precipitate to grow in perfect coherence with the decorated GB plane. In contrast, at the {130} boundary the precipitate retains double-Al-layer defects even after many MC swaps. These computational predictions rationalize that large Mg-rich phases form at each {120} facet, while the {130} facets experience only local segregation enrichment.

We also evidenced that the self-consistent co-evolution of facet structure and chemistry leads to the formation of periodic segregation patterns of 5 – 10 nm at Σ11, and Σ13a GBs [1]. Although the character of individual GB is often overlooked in the segregation study in engineering Al alloys, our study enriches the current understanding of the chemical-structural interactions. The solute segregation and heterogeneous precipitation at GBs have an impact on the corrosion properties of Al alloys. Within the same GB, the facets coupled with the anisotropic segregation and the precipitation behaviour will significantly influence the GB cohesion, intergranular fracture, and corrosion resistance of engineering Al alloys. Better predictions for the temporal evolution of the alloy's microstructure during aging and in service will require knowledge of the solute distribution within the microstructure, particularly at GBs.

References

1. Zhao, H.; Huber, L.; Lu, W.; Peter, N.; An, D.; De Geuser, F.; Dehm, G.; Ponge, D.; Neugebauer, J.; Gault, B.; Raabe, D.: Phys. Rev. Lett. 124 (2020) 106102.
2. Zhao, H.; De Geuser, F.; Kwiatkowski da Silva, A.; Szczepaniak, A.; Gault, B.; Ponge, D.; Raabe, D.: Acta Mater. 156 (2018) 318.
3. Sadigh, B.; Erhart, P.; Stukowski, A.; Caro, A.; Martinez, E.; Zepeda-Ruiz, L.: Phys. Rev. B 85 (2012) 184203.

Designing material properties by controlling impurity content in structural defects

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Controlling impurities in solid materials is a key to design materials' properties, putting doping at the core of modern materials technology. Increasing dopant concentration and reducing unwanted impurities in synthesised materials should make it possible to optimise the performance of target devices. This requires diverse research efforts, since establishing reliable design routes for materials containing desirable impurities requires a wide range of information ranging from atomistic insights to experimental validations. Several groups and departments at the MPIE joined forces to identify various mechanisms of impurities incorporation in solid materials by combining state-of-the-art experimental and theoretical approaches.

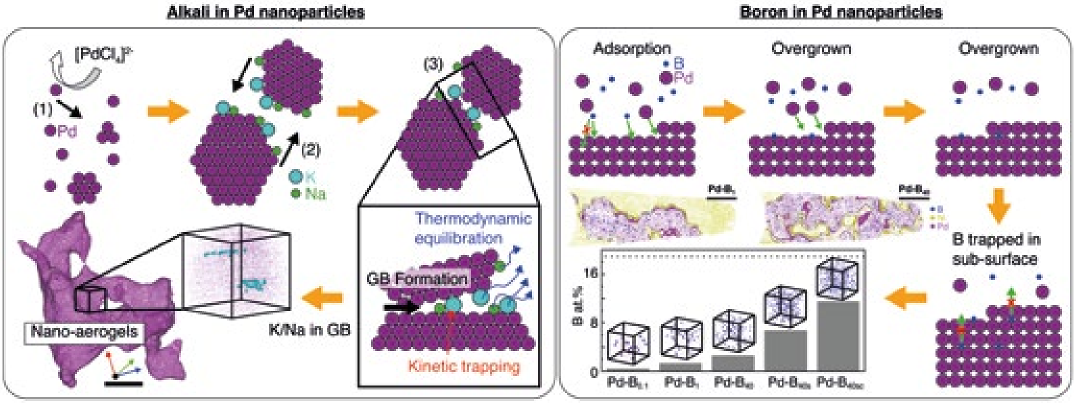

Fig. 1: *Schematic illustrations and experimental atom probe tomography (APT) validations of incorporations of impurities [alkali metals (left) and boron (right)] in Pd nanoparticles.*

Development for colloidal nanoparticles during the last 150 years allows materials scientists to synthesize nano-crystals, which are promising candidates for catalysis, by adding a reducing agent to a solution containing a metal precursor. Due to its excellent reducing properties, $NaBH_4$ is commonly used in the wet-chemical synthesis route for metal nanoparticles, since it can agglomerate to form a metallic nano-aerogel (MNA). These MNAs are often perceived as being purely metallic and free of any impurities. Yet, we directly observed the incorporation of alkali atoms (*i.e.*, Na, K) at the grain boundaries (GB) in Pd-MNAs.

To rationalize the alkali incorporation into the Pd-MNAs, we carried out a concentration analysis based on state-of-the-art density functional theory (DFT) calculations [1]. Using chemical reservoirs to account for the experimental conditions (*i.e.*, concentrations of reductant and precursor), we determine the thermodynamic equilibrium concentrations of alkali atoms in various geometric positions on the Pd surface, in the Pd bulk or in Pd GBs using their respective *ab initio* calculated energies. The comparison between theoretically calculated and experimentally observed concentrations of Na and K reveals that the comparatively larger K atoms are kinetically trapped at GBs. The smaller Na atoms achieve, in contrast, thermodynamic equilibrium during the coalescence of two nanoparticles, *i.e.*, the joining of two surfaces, as illustrated in the left panel of Fig. 1. S.-H. Kim was awarded for this work the E.W. Müller Outstanding Emerging Scientist Award at the Atom Probe Tomography & Microscopy 2021 virtual conference.

Given that our preliminary work showed that MNAs synthesized using $NaBH_4$ can no longer be considered pristine metallic nanostructures, we also measured the content of boron (B) incorporated in the Pd-MNAs from the $NaBH_4$ reductant and found it non-negligible. The B was found in the sub-surfaces region of the MNAs far from the surface [2]. Based on *ab initio* computed concentrations of B species on surfaces, the sub-surface layer and the bulk and taking into account the conditions during synthesis, the following scenario could be derived: B atoms adsorbed on the surface are kinetically frozen-in during the growth of MNAs by repeatedly overgrowing the top Pd surface layer by a subsequent Pd surface layer. Thus, the experimentally observed homogeneous B distribution is the consequence of non-equilibrium processes. As origin of this effect the substantially stronger binding energy of B in octahedral sites in the sub-surface region compared to on surface binding sites was identified: The sub-surface sites are at least 1 eV/B atom more favourable.

Having unravelled these incorporation mechanisms and considering that the Pd nanoparticles are promising materials of platinum-free catalysts for various catalytic reactions (*e.g.* the hydrogen oxidation reaction), we utilise our findings to suggest design routes for Pd-B nano-catalysts with different concentrations of B, by controlling the synthesis conditions, as shown in the right panel of Fig. 1.

It becomes quite obvious that an accurate determination of the impurity content in a sample is critical to understanding its properties. An omnipresent element, particular in an electrochemical environment, is hydrogen. This element is well known to affect materials properties. A well-known adverse phenomenon is H-embrittlement. Atom probe tomography (APT) is considered as one promising measurement technique to determine H contents in materials, due to its high spatial resolution and chemical sensitivity. However, there is a consensus that H-related species (*e.g.*, H^+, H_2^+ and H_3^+) are always produced under high-fields at the specimen's surface during APT measurements. The origin of H impurities is the subject of a long-standing debate. This indetermination limits our ability to precisely quantify H concentration in materials by APT. Advancing our understanding of the behaviour of H in the conditions of an APT experiment is hence absolutely essential. It will help elucidating numerous open questions in physics, chemistry, and materials science regarding H-involving mechanisms, including hydrogen trapping or grain boundary segregation of H in the context of hydrogen embrittlement.

To solve this long-standing debate of the APT community, we perform APT analysis on two very different metals - sodium (Na) and platinum (Pt). The sample preparation of alkali metals is challenging compared to transition metals, as their reactivity in contact with moisture and air leads to severe oxidation. So far, this has hindered the characterization of alkali by APT. Here, we used the specific setup of the Laplace Project to prepare and transfer specimens [3, see also p. xxx]. The mass spectrum of the first ever reported analysis of pure Na metal, interestingly, shows no H-related species. This contrasts with Pt that shows high H peaks, typical of the APT analysis of many metals, as shown in the middle inset of Fig. 2.

This result is counterintuitive: residual H_2 molecules in an APT vacuum chamber could be expected to ionize and dissociate due to the intense electric field and/or laser pulse near the apex and thus be detected regardless of the analysed material. To elucidate this phenomenon, we used calculations to carry out a thermodynamic analysis of Na and Pt surfaces in a H_2 gas environment and understand the stability of the metal surfaces under the given experimental conditions of pressure and temperature (shown in the right panel of Fig. 2). Our analysis indicates that Na surfaces have higher resistances to surface contamination with H compared to Pt surfaces, which can explain the absence of H-related peaks observed in the APT experiments of Na. Combining the theoretical analysis and considering different conditions encountered during APT experiments, specifically, specimen-preparation, transport and APT-operating conditions, we propose that the origin of H detected in APT measurements is primarily due to H contamination of the specimen's surface occurring during specimen preparation and transport or adsorption of residual background H_2 gas, as illustrated in the left panel of Fig. 2, and not ionisation of residual hydrogen in the chamber.

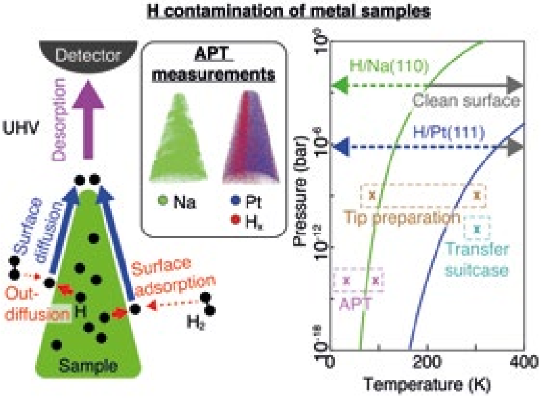

Fig. 2: *Schematic representation illustrating mechanisms of H contamination of transition metal samples (left). Experimental APT validations (in an inset) and theoretical analysis (right) to explain the experimental conditions of H contaminations.*

Our studies combining theoretical calculations, advanced characterization and catalytic activity measurements, have established the strength of 'impurity engineering' as a nanomaterial design strategy. Our collaborative activities focused on understanding the fundamental mechanisms of impurity incorporations into solid nanomaterials, which provides us with novel synthesis routes for tailoring properties of metallic structures by controlling the contents of impurities. Ongoing research [2] involving in addition the GO department will provide explicit insights into how controlling impurities in metallic nano-catalysts affects their catalytic performance for the hydrogen oxidation reaction (see p. 184).

References

1. Kim, S.-H.; Yoo, S.-H.; Chakraborty, P.; Jeong, J.; Lim, J.; El-Zoka, A.A.; Stephenson, L.T.; Hickel, T.; Neugebauer, J.; Scheu, C.; Todorova, M.; Gault, B.: J. Am. Chem. Soc. doi.org/10.1021/jacs.1c11680 (2022).
2. Kim, S.-H.; Yoo, S.-H.; El-Zoka, A.A.; Lim, J.; Jeong, J.; Schweinar, K.; Kasian, O.; Stephenson, L.T.; Scheu, C.; Neugebauer, J.; Todorova, M.; Gault, B.: in preparation.
3. Stephenson, L.T.; Szczepaniak, A.; Mouton, I.; Rusitzka, K.A.K.; Breen, A.J.; Tezins, U.; Sturm, A.; Vogel, D. ; Chang, Y.; Kontis, P.; Rosenthal, A.; Shepard, J.D.; Maier, U.; Kelly, T.F.; Raabe, D. ; Gault, B.: PLoS One 13 (2018) 1.
4. Yoo, S.-H.; Kim, S.-H.; Woods, E.; Gault, B.; Todorova, M.; Neugebauer, J.: arxiv.org/abs/2109.14981 (2021).

Hydrogen in iron alloys: from diffusion to mechanics

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Hydrogen embrittles high-strength structural materials, and its consequences are particularly important in steels, which are widely used in the automotive, aeronautical, gas, oil and power generation industries. The harmful effects of hydrogen in steels have been known for more than a century and, despite of numerous studies, the underlying mechanisms are still not well understood. One of the reasons is the elusive behaviour of hydrogen: its small dimension and high mobility render difficulties to directly image it and track its effects. Another important reason lies in the existence of a large variety of lattice defects that can interact with hydrogen and the complex nature of such interactions. The study of hydrogen in iron alloys and steels is a topic shared by all departments at the MPIE and these cooperative research activities fall into the three most important categories in the hydrogen embrittlement research:

1. hydrogen trapping and transport in complex microstructures;
2. quantitative measurement of local mechanical response under defined hydrogen activity;
3. hydrogen embrittlement micromechanisms and hydrogen tolerant microstructure design.

Experiments and computational simulations are carried out in model alloys and more complex steels.

A particular challenge is the study of diffusible or lightly trapped hydrogen and its effects on the material properties. A representative case study is ferritic Fe-Cr model alloys. In the defect-free lattice they show a high hydrogen mobility and low solubility; however, the introduction of additional elements, dislocations or grain boundaries changes significantly the alloy interactions with hydrogen. The concentration of hydrogen preserved in Fe-Cr alloys with different Cr content, grain size, and dislocation densities was quantified by thermal desorption spectroscopy (TDS). The alloys with a higher Cr content, higher dislocation density or smaller grain size can retain higher amount of hydrogen upon the same hydrogen charging conditions. Permeation studies were also started by using the novel Kelvin probe-based potentiometric hydrogen electrode method. As chromium, dislocations and grain boundaries are all effective trapping sites for hydrogen, the time lag for hydrogen passing through the alloy, in first step experiments, was found to increase, indicating more trapped hydrogen, while the rate also decreased. Further research is required to verify a comparable hydrogen activity and the effect on the diffusion coefficient. The capacity for permeation measurements will be soon increased (see p. 59).

The diffusivity and amount of hydrogen in the alloys have strong consequences in the mechanical behaviour. The alloy properties will be also affected by the specific location of hydrogen, which is measured by atom probe tomography and Kelvin probe experiments. Nanoindentation studies using an innovative setup for back-side hydrogen charging during mechanical testing [1] show, for instance, a reduction of the pop-in load (Fig. 1, left) and a hardening effect with increased hydrogen ingress into the referred Fe-Cr alloys (see p. 170). The Young's modulus is not affected by the introduced hydrogen. The effect of hydrogen is yet more pronounced in the alloys with higher Cr content. In single crystal and dislocation-free Fe-Cr alloys a linear relationship between the increase in hardness and the hydrogen supply was established. This hardening effect can be attributed to an enhanced dislocation multiplication (reduction of the pop-in load), pinning on the dislocation motion and an increase in lattice friction due to hydrogen.

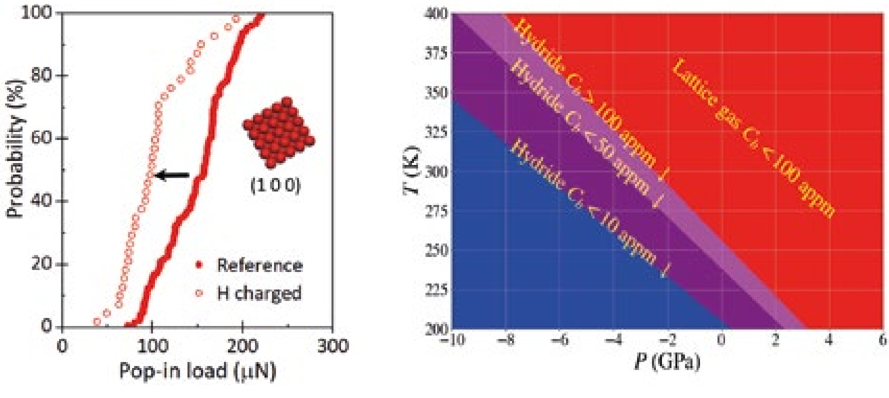

Fig. 1: *(Left) Probability plot of pop-in load during nanoindentation of Fe-20 wt.% Cr with and without hydrogen on a grain with (1 0 0) grain orientation. (Right) Pressure dependent phase diagram of the* Fe_4CrH_5 *hydride.* C_b *is the bulk concentration of hydrogen.*

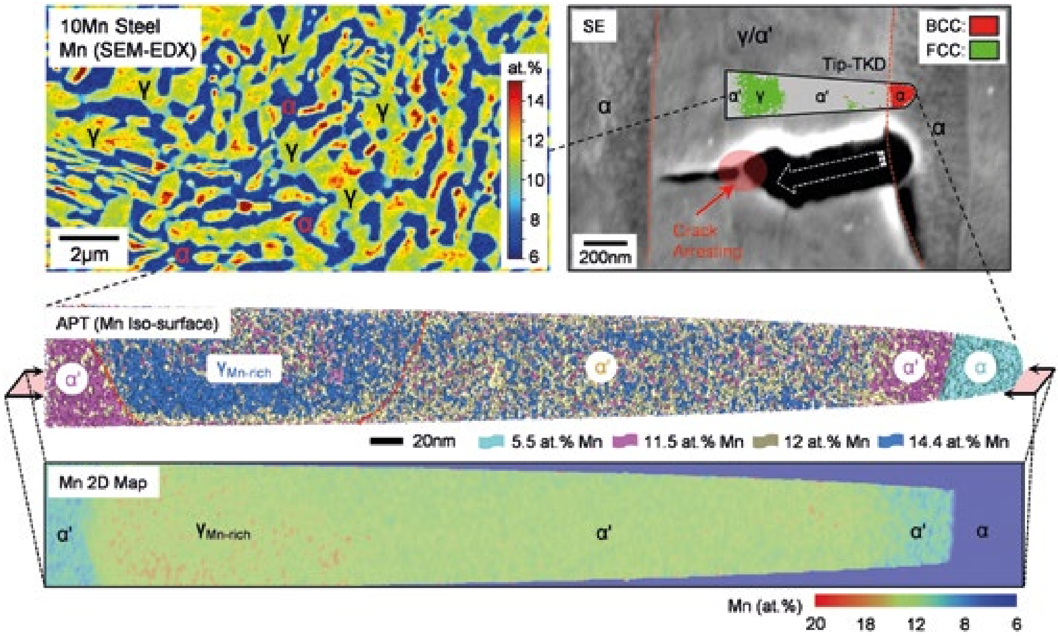

Fig. 2: *Chemically heterogeneous microstructure produced in a ferrite-austenite two phase medium Mn steel and the arresting of hydrogen-induced cracks by locally enriched Mn region which serves to locally stabilize austenite against martensite transformation.*

The reduction in the pop-in load due to the presence of hydrogen in metals is observed in the *in situ* nano-indentation experiments in various metals. The mechanism behind this reduction is not yet clear. In the case of the Fe-Cr alloys, using *ab initio* simulations, we observed attractive H-H interactions facilitating the formation of the nano-hydrides (NH). The magnitude of this attractive interaction is absent in pure Fe, but increases by increasing the Cr content. However, as it is depicted in the simulated phase diagram of Fe_4CrH_5 in Fig. 1 (right), the formation of this hydride at experimentally relevant T-C_b domain requires significant negative pressure. This tension can be attained in the vicinity of pre-existing dislocations. The formation of NHs helps homogeneous dislocation nucleation by inducing significant shear stress stemming from the misfit strain of the NH and the mismatch in its elastic moduli with respect to the metal. This finding enables us to predict the reduction in the pop-in load in Fe-Cr alloys.

In parallel to the research on the hydrogen diffusion and interaction with dislocations in model alloys, we also revealed hydrogen-induced damage mechanisms in the practically relevant transformation-induced plasticity (TRIP) steels. The deformation-driven martensitic transformation (i.e. the TRIP effect) strongly deteriorates the materials' resistance against hydrogen embrittlement, due to the high strength of the fresh martensite phase and the transformation-induced hydrogen redistribution [2]. We showed by slow strain rate tests, that the hydrogen embrittlement resistance can be significantly increased (by a factor of two) through adding stable austenite dispersed within an ultrafine grained microstructure. We exploited this microstructure design concept in a medium Mn steel (0.2C–10Mn–3Al–1Si in wt.%) and produced microscopically confined Mn heterogeneity throughout the microstructure (Fig. 2) [3]. The austenite is stabilized in the Mn enriched zone, which serves to stop hydrogen-induced cracks by blunting. On the other hand, the Mn-lean regions provide the required TRIP effect and thus a high strength-ductility combination. Such tailored microstructures with Mn heterogeneity were produced by a two-step annealing, which can be scalable to established and affordable industrial processing routes. Also, our new microstructure approach can be generalized to other types of high-strength steels with the same aim, namely, to lend their microstructures enhanced resistance against hydrogen embrittlement.

The studies of model iron alloys and complex steels lead to a better understanding of their interaction with hydrogen. This knowledge is essential in the design of hydrogen tolerant materials, required as structural alloys and for the growing use of hydrogen as energy source.

References

1. Duarte, M.J.; Fang, X.; Rao, J.; Krieger, W.; Brinckmann, S.; Dehm, G.: J. Mater. Sci. 56 (2021) 8732.
2. Sun, B.; Krieger, W.; Rohwerder, M.; Ponge, D.; Raabe, D.: Acta Mater. 183 (2020) 313.
3. Sun, B.; Lu, W.; Gault, B.; Ding, R.; Makeni, S. K.; Wan, D.; Wu, C.-H.; Chen, H.; Ponge, D.; Raabe, D.: Nature Mater. (2021) https://doi.org/10.1038/s41563-021-01050-y

Hydrogen interplay with defects in Al alloys

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The investigation of hydrogen in metallic alloys is a long standing key topic at the MPIE. While multiple insights on hydrogen embrittlement in steels has been gained over the last decades, similar phenomena in Al alloys have only recently come into focus, triggered by the ERC-funded SHINE project (see p. 50). Previously, Al alloys were often considered to be less affected by hydrogen embrittlement (HE), due to their superior corrosion resistance, the high activation energies for H incorporation and the low H solubility in the fcc matrix. This low solubility however can locally lead to large H concentrations at lattice defects and precipitates. Also, the increasing strength levels of modern Al alloys promote HE effects. For instance, in the high-strength 7xxx alloy series, where Zn, Mg, and Cu are main alloying elements, H-assisted crack initiation has become an issue of major concern. Three important key results connected to this phenomenon are highlighted in this report.

Competition of microstructure features in 7xxx Al alloys

The high strength of 7xxx Al alloys is achieved by its complex microstructure, containing not only grain boundaries (GBs), dislocations, micro-sized intermetallic phases but also a high dispersion of nanosized precipitates. For improving the materials' H-resilience to HE, it is important to determine for these features their respective potential for H trapping and crack initiation. Experimentally this is a highly challenging task, due to the requirement of combining highest spatial and chemical resolution with the probing sensitivity to detect individual hydrogen atoms.

The recent developments in cryo-atom probe tomography (APT) in the MA department, enabled by a completely controlled ultra-high vacuum (UVH) and cryogenic workflow consisting of charging, sample preparation and APT probing, using cryo-focused-ion beam techniques, makes it now possible to overcome this challenge. The charging of an Al-6.22Zn-2.46Mg-2.13Cu-0.155Zr (wt.%) was performed with deuterium (D), to distinguish it from residual hydrogen in the APT chamber [1]. The analysis of the data indicates a clear enrichment of D at the GB of the sample (Fig. 1b). The details of the complex GB is shown in Fig. 1c, which contains Al_3Zr dispersoids, 20-50 nm-sized (Mg, Zn)-rich precipitates, and also solutes. The compositional analysis shows that Al_3Zr-dispersoids at the GB (Fig. 1d) contain 11 at.% H and 0.6 at.% D. No enrichment in H and $D(H_2^+)$ is shown in (Mg, Zn)-rich precipitates distributed at the GB (Fig. 3e). The locally increased content of $D(H_2^+)$ implies that the Mg-decorated GB (i.e. devoid of precipitates) acts as a trap for H (Fig. 1f). A closer analysis is, however, required to explain why the experimentally observed crack initiates at GBs.

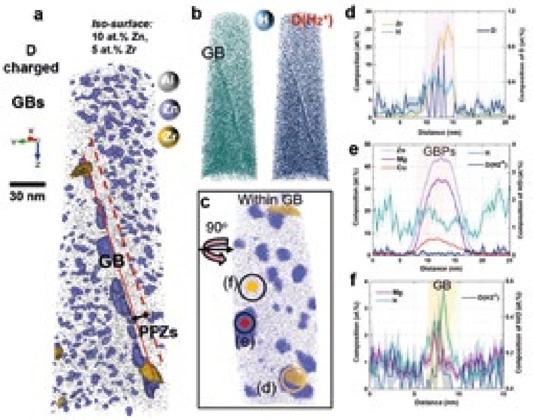

Fig. 1: *APT investigation of an Al-Zn-Mg-Cu tip that was charged with deuterium (D). The sample contains a grain boundary and a lot of Al_3Zr dispersoids, which are coarsened at the GB. Subfigure F displays a region of the GB without dispersoids. Adapted from [1].*

Therefore, density functional theory (DFT) calculations in the CM department have been used to achieve further insights into H segregation and HE. They indicate a much larger solubility of H in the dispersoids (provided Zr anti-sites are present) as compared to the GBs. It is, however, important to note that the absolute value of the embrittling energy, i.e., the H impact on the thermodynamic driving force for a decohesion of interfaces, shows a completely different trend. Despite the higher hydrogen concentration, the embrittling energy for the particles is very small. In contrast to this, the embrittling energy is larger for GBs (Fig. 2).

Effect of alloying elements on H embrittlement

The second interesting phenomenon concerns the impact of solutes on the embrittling energy. In Fig. 2 the effect of Mg is highlighted, because an enrichment of Mg next to GBs was also observed in APT (Fig. 1). The DFT calculations reveal hardly any change in the H segregation energy to the GB if Mg atoms are additionally present. However, the impact of even minor traces of solute Mg on the embrittling energy is still rather large. This is due to the highly attractive interaction of Mg and H at the new surfaces that form during crack initiation.

Due to these important atomistic observations the interaction of H with GBs in Al alloys has been studied more systematically. After extending the study to several types

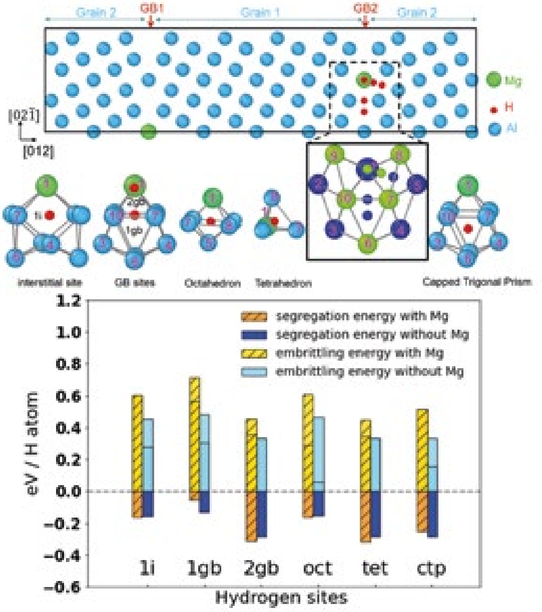

Fig. 2: *DFT calculations for the segregation and embrittling energy of H in a Σ5 (210) GB. The analysis has been performed for different interstitial sites in the GB, comparing a scenario with and without Mg atoms nearby the H sites. Adapted from [1].*

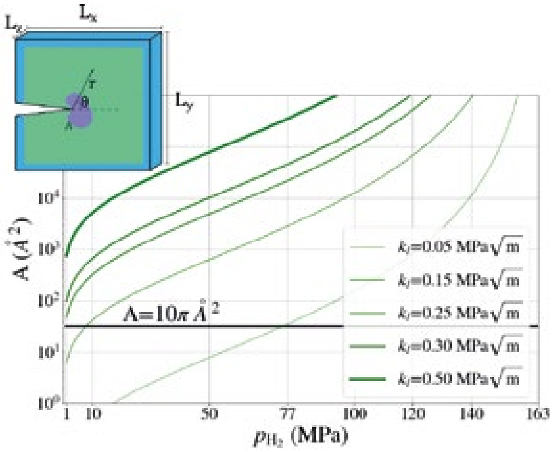

Fig. 3: *Distribution of the cross-sectional area of the hydrides formed around the crack tip at T = 300 K versus the H_2 pressure for different values of the cracks stress intensity factor. The schematic diagram for an Al alloy containing a local hydride (purple) next to a crack tip is also shown.*

of GBs, we were able to correlate the solution enthalpy to a relative change in the Voronoi volume and to generalize the finding to a model for the solution enthalpy. This will allow a prediction of the solution enthalpy even for GBs that are too complex to be treated by DFT calculations.

The extension to several alloying elements relevant for the 7xxx series of Al alloys reveals that the behaviour of Mg is not unique, but that solute Sc and Zr have a qualitatively similar and even larger impact on the H embrittlement. The solute atom Sn, however, turns out to be highly interesting, because it strongly binds with H in the GB, without increasing the embrittling tendency. Based on these insights, effective alloying strategies to improve the resistance to HE can be developed.

Impact of the microstructure on hydride formation

The difference described above between the hydrogen solubility in the bulk phase and in the vicinity of GBs already indicates a strong impact of the microstructure. If cracks form, this can go so far that hydride formation becomes possible. Due to the relevance for hydrogen storage applications, the Al hydride formation has, therefore, been further investigated as prototypical example of a metallic hydride former enhanced by defects.

To this end, DFT calculations have first been used to determine the formation enthalpy of the most common Al hydrides in their pure bulk phases as a function of the H chemical potential and a negative mechanical pressure in the system. The former can be modified by the external H_2 gas pressure, whereas the latter is subject to an external load. These calculations reveal that a negative pressure field of magnitude −4 GPa significantly reduces the formation energy of α-Alane hydride, but is negligible for the other hydride phases.

The presence of negative pressure fields at a crack tip is well-known. It is best described by the stress-intensity factor, which itself is proportional to an external load. Hence, by the application of a tensile strain to the sample, a control of the stress intensity factor and, therefore, of the formation of local hydrides becomes feasible. In order to demonstrate this effect, the formation energy of the Alane hydride is modified by including the interaction of its excess volume and the stress field of a tensile crack. The schematic diagram of the hydride and crack as well as the enhancement in formation of the hydride are illustrated in Fig. 3. In the absence of an external load, an unrealistically high hydrogen gas pressure of 163 MPa would be required in order to form a hydride phase. If a k-load of ≈0.15 MPa√m is applied, a hydride with a critical size for the cross-sectional area is already formed with a gas pressure of 10 MPa. The value should be compared with the critical k-load for dislocation emission next to cracks, which is about 0.3 MPa√m. The findings need to be critically balanced against the failure of the material due to brittle fracture and HE, but the potential of defects for the formation of metallic hydrides becomes evident from this study.

References

1. Zhao, H.; Chakraborty, P.; Ponge, D.; Hickel, T.; Sun, B.; Wu, C.-H.; Gault, B.; Raabe, D.: Nature (2021) accepted.

Near-atomic-scale analysis of hydrides

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Hydrides are hydrogen-containing stable phases. Most hydrides are mechanically brittle and their formation associated to hydrogen ingress in materials over the course of their service life is a major contributor to the 'hydrogen embrittlement' of entire classes of engineering alloys. Titanium and zirconium are known bulk hydride formers, and their alloys are often subject to hydride-related failure. The detailed study of their formation mechanisms remains highly challenging. Scanning and transmission-electron microscopy (S & TEM) and associated techniques require dedicated specimen preparation protocols for reliably revealing hydride formation, since hydrogen can be introduced into the specimen by the sample preparation alone. We have developed methodologies that enable to differentiate between hydrides formed in the sample during preparation vs. those stemming from the preceding environmental exposure by for example hydrogen charging. Although atom probe tomography (APT) is known to be capable of 3D compositional mapping with sub-nanometre resolution for a wide range of materials, there are very few studies of hydrides and solute hydrogen analysis remains an absolute frontier [1]. Here we set ourselves to investigating the detailed formation mechanisms of hydrides in some zirconium and titanium alloys.

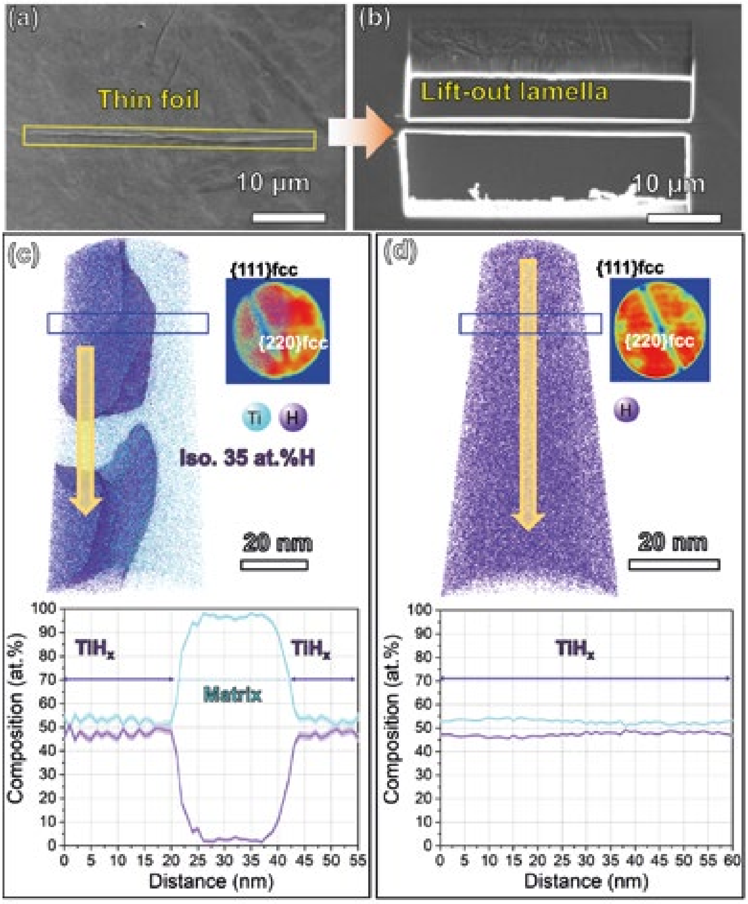

Fig. 1: *Semi-correlative APT analysis of fcc-hydrides in the electropolished TEM specimen. (a) and (b) are SEM images of the site-specific APT sample preparation of a lenticular hydride. (c) and (d) show 3D atom maps, detector histograms and 1D concentration profiles. The detector histograms and concentration profiles are extracted from the blue rectangle and yellow arrow, respectively. (c) Concentration profile across two matrix-hydride interfaces extracted across the yellow arrow. The ion detection map shows a {220}fcc pole. (d) 3D atom map and 1D concentration profile within the hydride.*

In titanium and its alloys, we first demonstrated the possibility to measure hydrogen content and partitioning between the different phases, and analyse hydrides by APT [2]. We discussed the importance of alloying to avoid hydride formation, too. However, the hydrogen-content was ill-controlled, coming from the specimen preparation, and we then demonstrated the importance of using cryogenic-temperature during the final stage of specimen preparation to avoid the introduction of spurious hydrogen and hydrides [3], as well as our capacity for forming and studying Ti-hydrides by APT [4]. Finally, we combined high-resolution TEM with electron-energy loss spectroscopy and APT (Fig. 1) to demonstrate that a phase that is sometimes identified as a face-centred cubic allotrope of Ti is, in fact, simply a hydride formed during the preparation of the specimens [5].

As part of the ERC Consolidator Grant Shine project (see p. 50), a collaboration was established with colleagues at Imperial College London (group of T.B. Britton, now at University of British Columbia). They used electrochemical charging of Zr-alloys to introduce high concentrations of hydrogen or deuterium, and by adjusting the cooling rate following a homogenisation heat treatment, provided samples with hydride at different stages of growth. The study of both hydrides and deuterides formed under similar conditions allowed us to optimise the analysis conditions and ensure reliable compositional analyses [6]. In slow cooled samples, we reported on the partitioning of Sn away from intergranular hydrides, with a strong pile-up at the interface, in the vicinity of which metastable interfacial hydride between the δ-hydride and the metallic matrix was detected [7]. We observed no redistribution of other major alloying elements (e.g. Fe) that appear mostly segregated to the GBs, but revealed that Sn is also found segregated to stacking faults forming ahead of the hydride growth front, because of the difference in lattice parameter between the hydride and the metallic matrix [8]. A follow-up study was performed on a fast-cooled sample, and demonstrated that the Sn partitioning already takes places from the early stage and that nucleation of hydrides can be aided by the presence of secondary-phase particles across the metallic matrix [9].

Importantly all these studies demonstrated the possibility of performing clean high-resolution imaging and quantification of hydrogen both as a solute and inside of different phases in these materials.

References

1. Breen, A.J.; Stephenson, L.T.; Sun, B.: Li, Y.; Kasian, O.; Raabe, D.; Herbig, M.; Gault, B.: Acta Mater. 188 (2018) 108.
2. Chang, Y.; Breen, A.J.; Tarzimoghadam, Z.; Kürnsteiner, P.; Gardner, H.; Ackerman, A.K.; Radecka, A.; Bagot, P.A.J.; Lu, W.; Jägle, E.A.; Herbig, M.; Stephenson, L.T.; Moody, M.P. Rugg, D.; Dye, D.; Ponge, D.; Raabe, D.; Gault, B.: Acta Mater. 150 (2018) 273.
3. Chang, Y.; Lu, W.; Guénolé, j.; Stephenson, L.T.; Szczpaniak, A.; Kontis, P.; Ackerman, A.K.; Dear, F. F.; Mouton, I.; Zhong, X.; Zhang, S.; Dye, D.; Liebscher, C.H.; Ponge, D.; Korte-Kerzel, S.; Raabe, D.; Gault B.: Nature Comm. 10(1) (2019) 942.
4. Chang, Y.; Mouton, I.; Stephenson, L.T.; Ashton, M.; Zhang, G.; Szczpaniak, A.; Lu, W.; Ponge, D.; Raabe, D.; Gault, B.: New J. Phys. 21 (2019) 053025.
5. Chang, Y.; Zhang, S.; Liebscher, C.H.; Dye, D.; Ponge, D.; Scheu, C.; Dehm, G.; Raabe, D.; Gault, B.; Lu, W.: Scripta Mater. 178 (2020) 39.
6. Mouton, I.; Breen, A.J.; Wang, S.; Chang, Y.; Szczepaniak, A.; Kontis, P.; Stephenson, L.T.; Raabe, D.; Herbig, M.; Gault, B.: Microsc. Microanal. 25(2) (2019) 481.
7. Breen, A. J..; Mouton, I.; Lu W; Wang, S.; Szczepaniak, A.; Kontis, P.; Stephenson, L.T.; Chang, Y.; Kwiatkowski da Silva, A.; Liebscher, C.H.; Raabe, D.; Britton, T.B.; Herbig, M.; Gault, B.: Scripta Mater. 156 (2018) 42.
8. Mouton, I.; Chang, Y.; Chakraborty, P.; Wang, S.; Stephenson, L.T.; Britton, T.B.; Gault, B.: Materialia 15 (2021) 101006.
9. Lu, W.; Kontis, P.; Wang, S.; Birch, R.; Wenman, M.; Gault, B.; Britton, T.B.: arXiv:2109.10955.

Mn segregation at Fe grain boundaries

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Grain boundary segregation in metallic alloys can be connected with important phenomena [1] such as spinodal decomposition, heterogenous phase nucleation, suppressed coarsening, embrittlement and crack initiation. Due to their advanced mechanical behaviour, high- and medium Mn steels belong to the most important metallic alloys studied at the MPIE within the last decades. Due to the dual benefit of scientific novelty as well as industrial relevance, the observation of spinodal fluctuations at grain boundaries in Fe-Mn alloy by the MA department in 2018 [2] has initiated intensive research at the MPIE in the last years.

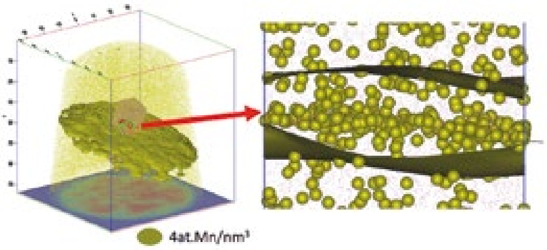

Fig. 1: *Detail of a monolayer of Mn atoms segregated at a GB in a Fe-4 at%Mn alloy aged during 2 months at 450 °C. Mn atoms are represented as yellow spheres and iron atoms as pink dots.*

Figure 1 shows such an atom probe tomography (APT) analysis of a grain boundary enriched with Mn in a Fe-4 at%Mn alloy. The segregation of the Mn atoms to the grain boundary (GB) is clearly highlight by the iso-concentration profile. In similar experiments with Fe-9 at.% Mn alloys [2] it was shown that the enrichment can yield the nucleation of an austenitic phase at the GB with a composition of 32 at.% Mn. Combining the chemical information with thermodynamic modelling, spinodal decompositions and the formation of defect phases (see p. 192) have been discussed in this context.

The underlying physical mechanisms are a result of highly interesting coupling phenomena: First of all, in order to achieve a grain boundary engineering, a series of thermal treatments need to be performed in experiments. This does not only yield the above-described chemical segregation. During this process, also the magnetic properties in the vicinity of the grain boundary can be altered. In high temperature heat treatments, the magnetic entropy supports the magnetically disordered state (or paramagnetic state) in the material. However, the link between magnetic states (particularly the paramagnetic state) and segregation is unexplored in the literature, due to the theoretical and computational complexities in handling magnetically disordered state in particular in the presence of the structural complexity imposed by grain boundaries. Here, some methodological achievements in this regard are highlighted.

Segregation in magnetically ordered and disordered states

First-principles density functional theory (DFT) based approaches are adopted to determine segregation profiles in the magnetically ordered ferromagnetic state and disordered paramagnetic states (see p. 71). Within the adiabatic approximation, since the magnetic degrees of freedom are much faster than the atomic ones, a spin-space average (SSA) based relaxation scheme [3] is developed to compute energies in the paramagnetic state. The segregation energies for Mn at different sites in the Σ5(310) grain boundary are presented in Fig. 2.

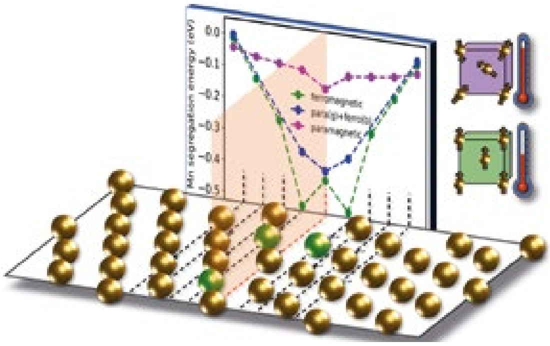

Fig. 2: *Schematic of the Σ5(310) grain boundary with dashed lines indicating layers next to the GB layer (red line). The diagram shows the segregation energies in the ferromagnetic and paramagnetic states. Green/golden balls indicate Mn/Fe atoms.*

The negative values of segregation energy in Fig. 2 indicate favourable segregation. The segregation drive is large in the ferromagnetic state. This is not only a thermodynamic observation, but also kinetic *ab initio* studies show a higher ratio of the Mn/Fe diffusion rate in the ferro- as compared to the paramagnetic state [4]. Further, the sites in the layer next to the grain boundary in the ferromagnetic state turn out to be the most favourable. This is counter-intuitive, since one would expect the site in the grain boundary layer to be most favourable, because of the larger size of the Mn atoms. To explain this, we show magnetic interaction parameters and segregation energies of antiferromagnetic Fe atom in the ferromagnetic environment in Fig. 3.

Here, a larger value of magnetic interaction parameter at a particular site indicates that incorporation of an antiferromagnetic Fe atom at that site is more unfavourable. Clearly, the lowest value at site 2 confirms that the preference of Mn at this site is mainly due to its antiferromagnetic nature. The presence of an antiferromagnetic atom at this site reduces the magnetic repulsion between two otherwise closely situated ferromagnetic atoms.

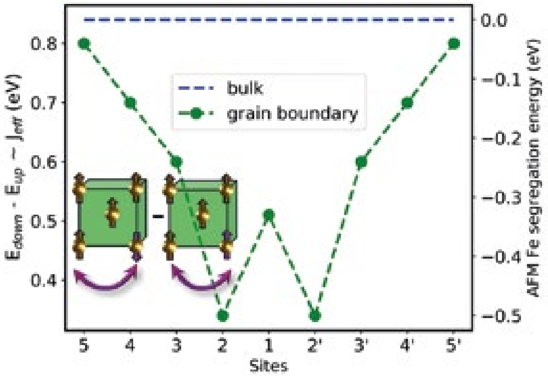

Fig. 3: *Magnetic interaction parameters at different sites of the grain boundary. The corresponding segregation energies of antiferromagnetic Fe atoms are shown in the second y-axis.*

At the same time, the result in Fig. 3 can also be interpreted as segregation profile for an antiferromagnetic Fe atom, with a segregation energy shown in the secondary *y*-axis. Interestingly, this profile is qualitatively and quantitatively very similar to that of the Mn segregation in Fig. 2. Finally, the absence of this feature in the paramagnetic state, where the magnetic moments are random by default and the Mn moment is not special, further confirms that the segregation of Mn at Fe grain boundaries is largely driven by magnetic interactions. Therefore, the segregation drive in the paramagnetic state is significantly smaller as compared to the ferromagnetic state.

Magnetic, chemical, and structural interplay at the grain boundary

While the impact of magnetic states on segregation profiles is discussed above, question arises as to whether the formation of grain boundary and Mn segregation could in turn affect the local magnetic state. Computing the magnetic transition temperature from the ferromagnetic to paramagnetic state, i.e., the Curie temperature, helps in classifying the magnetic state at any given temperature. Therefore, Curie temperatures calculated for both the bulk and grain boundary as a function of Mn concentration are shown in Fig. 4. Since the mean-field definition overestimates the bulk bcc Fe Curie temperature, the rescaled values (by the corresponding factor) are shown in the secondary *y*-axis. The figure conveys two remarkable messages: (1) The Curie temperature substantially reduces by the presence of a grain boundary. (2) The accumulation of Mn at the grain boundary results in further reduction in the Curie temperature with a rate of 12 K/at.%. Due to both effects, there exists a temperature window where the grain boundary exhibits the paramagnetic state whereas the bulk is ferromagnetic. Thus, the figure reveals an impressive coupling of structure, chemistry, and magnetism at FeMn grain boundaries. The direct access to Curie temperature facilitates the determination of the magnetic state of the grain boundary at any given temperature, the knowledge of which can be used to derive the segregation energies and occupancies pertaining to that magnetic state at the given temperature. In this way, one can provide new opportunities for segregation engineering in experiments based on quantitative prediction from computations.

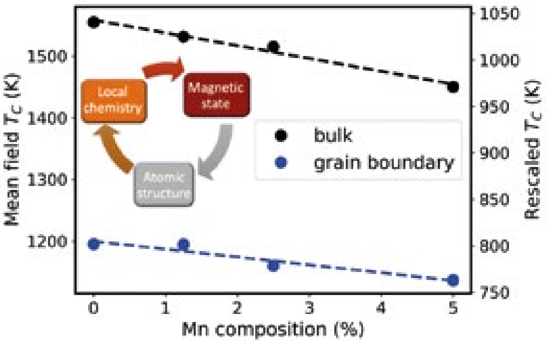

Fig. 4: *Mean field Curie temperatures for the grain boundary structure and the bulk bcc structure with respect to Mn concentration. The secondary y-axis shows the Curie temperatures rescaled to the experimental value.*

References

1. 1. Raabe, D.; Herbig, M.; Sandlöbes, S.; Li, Y.; Tytko, D.; Kuzmina, M.; Ponge, D.; Choi, P.-P.: Curr. Opin. Sol. Stat. Mater. Sci. 18 (2014) 253.
2. 2. Kwiatkowski da Silva, A.; Ponge, D.; Peng, Z.; Inden, G.; Lu, Y.; Breen, A.; Gault, B.; Raabe, D.: Nature Comm. 9 (2018) 1137.
3. 3 Hegde, O.; Grabowski, M.; Zhang, X.; Waseda, O.; Hickel, T.; Freysoldt, C.; Neugebauer, J.: Phys. Rev. B 102 (2020) 144101.
4. 4. Hegde, O.; Kulitckii, V.; Schneider, A.; Soisson, F.; Hickel, T.; Neugebauer, J.; Wilde, G.; Divinski, S.; Fu, C.-C.: Phys. Rev. B 104 (2021) 184107.

Interstitials in high entropy alloys

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Interstitial alloying in high-entropy alloys (HEAs) is an important strategy for tuning and improving their mechanical properties. Strength can be increased due to interstitial solid-solution hardening, while interstitial alloying can simultaneously affect, e.g., stacking fault energies (SFEs) and thus trigger different deformation mechanisms. While this provides a variety of opportunities, it introduces at the same time a further degree of complexity. An understanding of the fundamental mechanisms on the atomic scale is therefore crucial.

For HEAs, each interstitial site displays a different local environment and thus a specific solution energy (Fig. 1). Resolving such complex local configuration dependencies is usually not accessible to experiments alone. We therefore investigated the impact of interstitials (C and N) on the equiatomic CrMnFeCoNi HEA based on first-principles calculations [1, 2]. Both, the face-centred cubic (fcc) and the hexagonal close-packed (hcp) phases of CrMnFeCoNi were modelled based on the supercell approach to evaluate SFEs. The solution energies of interstitial C and N atoms are computed for both phases. A large number of interstitial sites are investigated to elucidate the dependence on the local environments around the interstitial atoms. By scanning over various chemically different local environments, a highly realistic representation of the stability and the energetics of interstitials in CrMnFeCoNi could be obtained.

Interestingly, the distribution of the interstitial C solution energies (Fig. 1) shows a width of more than 1.5 eV, which is an order of magnitude larger than the thermal energy at relevant annealing temperatures (approximately 0.1 eV at 1000 K). This large variation of solution energies also indicates a strong dependence of them on the local environment of C. Utilizing different thermodynamic statistical treatments it is found that both, C and N, increase the SFEs [1, 2]. This is consistent with our experimental observations that interstitial C increases the stability of the fcc phase in a metastable non-equiatomic FeMnCoCrC alloy [3]. Our analysis of local correlations revealed further, that C in CrMnFeCoNi energetically prefers interstitial sites with lower local valence-electron concentration, suggesting segregation of C into Cr-rich environments, hence promoting Cr-rich carbides. Indeed, we experimentally found that Cr-rich $M_{23}C_6$ carbides can be found in the non-equiatomic FeMnCoCrC alloy after annealing [3]. Due to the slightly increased SFE and the trend to form Cr-rich carbides by introducing the interstitial element C, the non-equiatomic FeMnCoCrC alloy unifies all metallic strengthening mechanisms including precipitation strengthening, twinning- and transformation-induced plasticity, apart from the massive substitutional and interstitial solid solution strengthening, leading to twice the tensile strength compared to an interstitial-free HEA with similar composition, yet, at fully maintained ductility [3].

We have further extended the alloy design concept by interstitial alloying to nanostructure HEAs thereby increasing their strength and high temperature stability. This nanostructuring was explored in a model $Fe_{19.84}Mn_{19.84}Co_{19.84}Cr_{19.84}Ni_{19.84}C_{0.8}$ (at.%) iHEA processed by cold-rolling and subsequent annealing at 800 °C and 900 °C [4].

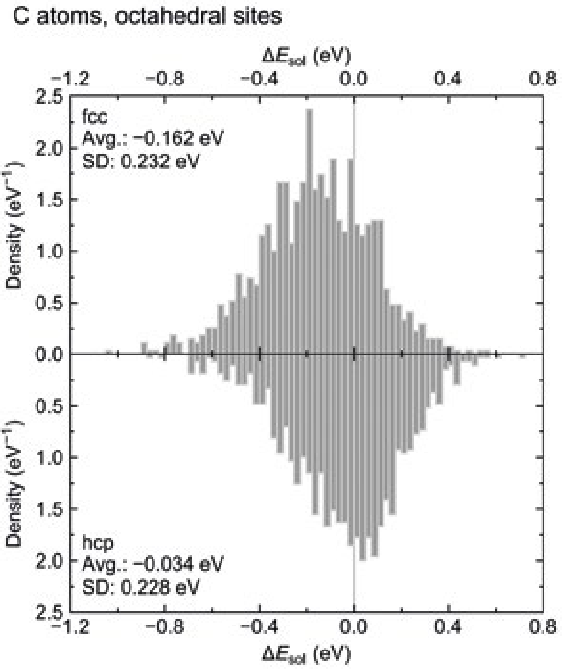

Fig. 1: *Distribution of computed solution energies of interstitial C atoms at the octahedral sites in CrMnFeCoNi. The upper and the lower panels show the results for the fcc and the hcp phases, respectively. The average (avg.) and the standard deviation (SD) of the solution energies are also shown in the panels, revealing that C increases the stacking fault energy (fcc – hcp stability).*

After the annealing treatment at 900 °C the microstructure of the iHEA is characterized by recrystallized and nano-twinned regions. A deep microstructural characterized by scanning / transmission electron microscopy (S/TEM) and atom probe tomography (APT) revealed that elongated $M_{23}C_6$-type nanocarbides (Fig. 2) are forming

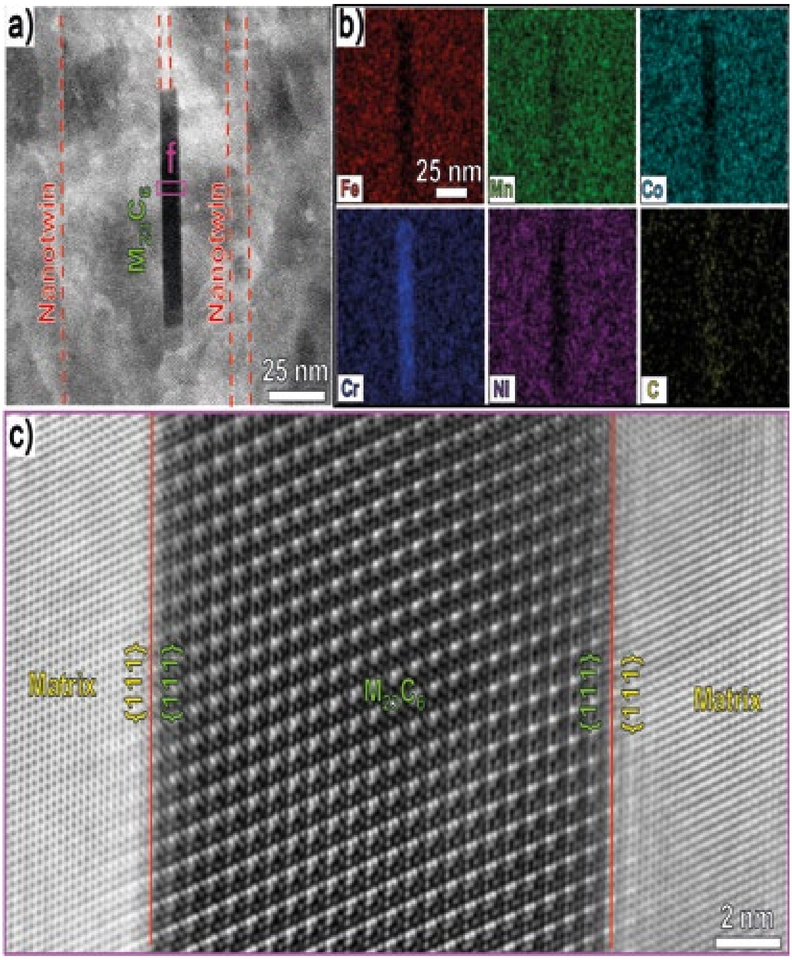

Fig. 2: *STEM analysis of elongated nanocarbides. (a) High-angle annuar dark-field imaging-STEM image of a nanocarbide that precipitated along nanotwin boundaries. (b) The corresponding STEM-EDS maps reveal a strong enrichment of Cr in the carbide. (c) Atomic structure of the elongated* $M_{23}C_6$ *(M: Fe, Mn, Co, Ni, Cr) by high-resolution STEM from the carbide shown in (a) revealing the orientation relationship between matrix and nanocarbide:* $<110>_{matrix}$ $//<110>_{nanocarbide}$ *and* $\{111\}_{matrix}//\{111\}_{precipitate}$.

along the nanotwins, stabilizing them even up to elevated temperatures of 800 °C. This heterogeneous microstructure leads to a significant increase in ultimate tensile strength of >1.2 GPa compared to an equiatomic FeMnCoCrNi HEA with ~700 MPa. It also provides outstanding high temperature mechanical properties resulting in yield strengths of ~650 MPa and ~300 MPa at 600 °C and 700 °C, respectively. The underlying mechanisms of the formation of the nanocarbides were explored by atomic resolution and *in situ* S/TEM. We found that nanoscale 9R structures are forming along the nanotwin boundaries before the annealing treatment. Their high dislocation content accelerates C diffusion and hence they serve as nucleation sites for the formation of the elongated $M_{23}C_6$-type nanocarbides. These nanophases stabilize the nanotwins enabling to overcome the trade-off between strength and thermal stability, thus qualifying these interstitially alloyed HEAs as promising pathway for the design of strong and ductile thermally stable bulk nanotwinned materials.

References

1. Ikeda, Y.; Tanak, I.; Neugebauer, J.; Körmann, F.: Phys. Rev. Mat. 3 (2019) 113603.
2. Ikeda, Y.; Körmann, F.: J. Phase Equilib. Diffus. 42 (2021) 551.
3. Li, Z.; Tasan, C. C.; Springer, H.; Gault, B.; Raabe, D.: Sci. Rep. 7 (2017) 40704.
4. Lu, W.; Liebscher, C.H.; Yan, F.; Fang, X.; Li, L.; Li, J.; Guo, W.; Dehm, G.; Raabe, D.; Li, Z.: Acta Mater. 185 (2020) 218.

Advanced compositionally complex alloys

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The exploration of high dimensional composition alloy spaces, where five or more alloying elements are added at near equal concentration, triggered the development of so-called high entropy (HEAs) or compositionally complex alloys (CCAs). This new design approach opened vast phase and composition spaces for the design of new materials with advanced mechanical and functional properties.

The development of alloys with tailorable strength and ductility was enabled by compositional tuning of FeMnCoCrNi alloys. By adjusting the Fe/Mn ratio it was possible to obtain single phase face centered cubic (fcc) alloys, which predominantly deform via nanotwining and dislocation plasticity, equipping them with an excellent strength-ductility synergy [1]. The key factor controlling the dominant deformation mechanism is rooted in the Mn content, which ultimately impacts the stacking fault energy as shown by density functional theory calculations. In dual-phase FeMnCoCr alloys a novel bidirectional transformation induced plasticity effect was observed, which explains the outstanding work-hardening capacity in these alloys [2]. To enhance the capabilities in mechanical properties to elevated temperatures, we also extended the alloy design concept to interstitial alloying (see p. 208) with C in FeMnCoCrNiC alloys [3]. In this way it was possible to stabilize nanotwinned regions by the precipitation of nano-carbides up to temperatures of 800 °C providing excellent mechanical properties for temperatures of up to 700 °C with a tensile yield strength of ~300 MPa.

Amorphous-crystalline nanocomposite structures have been found to promote an ultrahigh yield strength with large deformability. The alloy design concept was realized in a FeCoCrNiSiB high-entropy nanocomposite [4] and Cr-Co-Ni (crystalline, ~18 nm-thick)/TiZrNbHfCrCoNi (amorphous, ~12 nm-thick) laminate composite alloy [5], respectively. Furthermore, the laminate composite alloy exhibits ~200 K higher crystallization temperature (TX> 973 K) compared to that of the original TiZrNbHf-based amorphous phase. The elemental partitioning among adjacent amorphous and crystalline phases leads to their mutual thermodynamic and mechanical stabilization, opening up a new symbiotic approach for stable, strong and ductile materials. These design concepts were then adapted to develop wear-resistant metals, which achieve superior wear resistance via *in situ* formation of a strong and deformable oxide crystal-glass nanocomposite surface during wear in an oxidative environment – referred to as 'reactive wear protection' [6]. This reactive wear protection strategy offers a pathway for designing ultra-wear resistant alloys.

In an approach to also explore the potential of CCAs for magnetic applications we combined experimental investigations and theoretical calculations to unveil an abnormal magnetic behaviour caused by addition of the nonmagnetic element Cu in face-centred-cubic FeNiCoMn-based CCAs [7]. Interestingly, upon Cu addition, the CCAs show an increase of both Curie temperature and saturation magnetization in as-cast and homogenized states. Also, the saturation magnetization of the as-cast HEAs at room temperature increases by 77 % and 177 % at a Cu content of 11 and 20 at. %, respectively, compared to the initial, Cu-free as-cast equiatomic FeNiCoMn HEA. We found that the increase in saturation magnetization of the as-cast HEAs is associated with the formation of an Fe-Co rich phase. For the homogenized alloys, the magnetic state at room temperature transforms from a paramagnetic into a ferromagnetic one upon addition of 20 at.% Cu. Both, the increase of the saturation magnetization as well as the Curie temperature could not be adequately explained by the formation of Cu enriched zones according to our atom probe tomography (APT) analysis. Our *ab initio* calculations suggest that Cu plays a crucial role in the stabilization of a ferromagnetic ordering of Fe, and also reveal an increase of the Curie temperature caused by the Cu, a result which agrees well with the experimental findings. The underlying mechanism behind this phenomenon lies in a combined change in unit-cell volume and chemical composition and the related energetic stabilization of the magnetic ordering upon Cu alloying as revealed by our theoretical calculations. The possibility to improve the magnetic properties of HEAs by addition of nonmagnetic elements like Cu opens a great avenue to further exploring HEAs for magnetic applications.

An alternative route to improve the magnetic properties of multicomponent alloys has been to render a massive solid solution metastable and to trigger spinodal decomposition [8]. The motivation for starting from a HEA has been to provide the chemical degrees of freedom required to tailor spinodal behaviour using multiple components. The key idea is to form Fe-Co enriched regions which have an expanded volume (relative to a hypothetical unconstrained Fe-Co bulk), due to coherency constraints imposed by the surrounding HEA matrix. As demonstrated by our theory and experiments, this leads to improved magnetic properties of the decomposed alloy relative to the original solid solution matrix. In a pro-

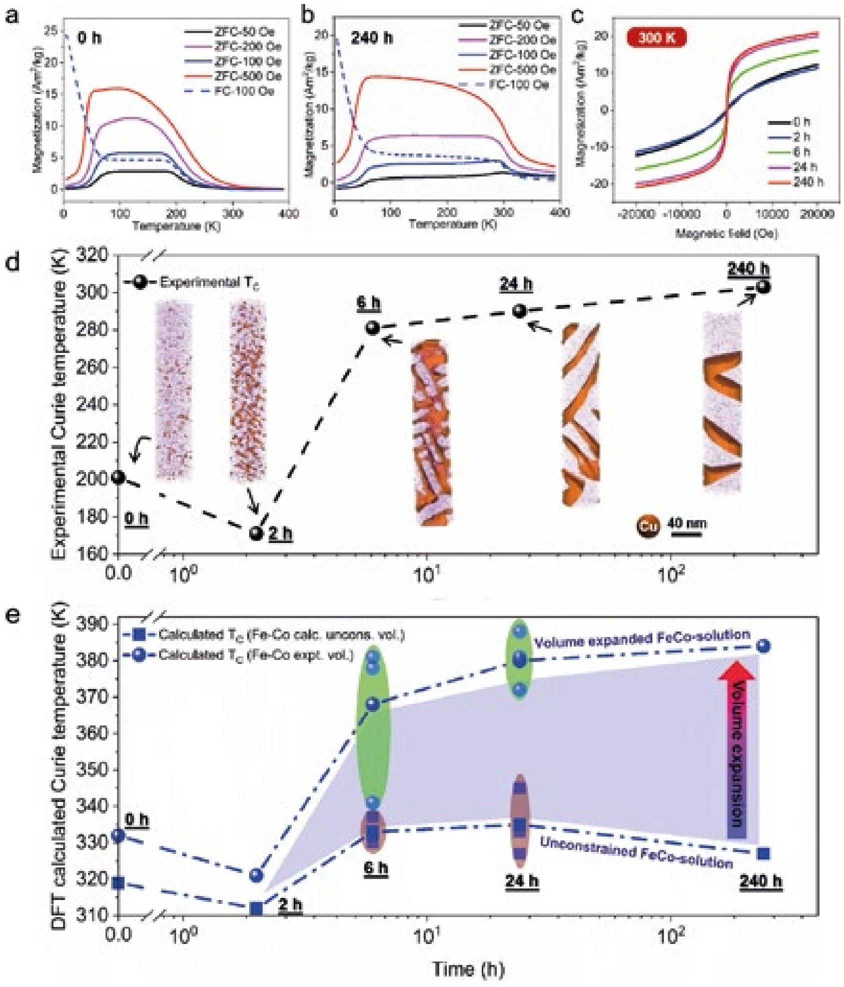

Fig. 1: *Magnetic properties of the FeCoNiMnCu HEA samples exposed to different processing conditions. a) Temperature dependence of magnetization of the homogenized and b) 600 °C/240 h annealed HEA samples. c) Hysteresis loops with different annealing time at 300 K. d) Experimental Curie temperatures as a function of annealing time. The morphological evolution of the alloy's nanostructure as a function of annealing time is shown in terms of APT reconstructions, showing also 50 at. % iso-composition surfaces of Cu. e) DFT calculated Curie temperatures as a function of annealing time. The blue shaded area indicates the impact of strain induced volume dilatation on the Curie temperature, i.e., the difference between the Curie temperature calculated for a hypothetical free-standing unconstrained single-phase bulk-like Fe-Co alloy and that for the experimentally measured strained volume of the same region in the HEA. Oval shapes indicate fluctuations due to scatter in local compositions (squares and circles) among different APT specimens.*

totype magnetic FeCoNiMnCu HEA, it is shown that the modulated structures, achieved by spinodal decomposition, lead to an increase of the Curie temperature by 48 % and a simultaneous increase of magnetization by 70 % at ambient temperature as compared to the homogenized single-phase reference alloy (Fig. 1). These findings open a pathway towards the development of advanced functional HEAs for magnetic applications.

References

1. Guo, W.; Su, J.; Lu, W.; Liebscher, C.H.; Kirchlechner, C.; Ikeda, Y.; Körmann, F.; Liu, X.; Xue, Y.; Dehm, G.: Acta Mater. 185 (2020) 45.
2. Lu, W.; Liebscher, C.H.; Dehm, G.; Raabe, D.; Li, Z.: Adv. Mater. 30 (2018) 1804727.
3. Lu, W.; Liebscher, C.H.; Yan, F.; Fang, X.; Li, L.; Li, J.; Guo, W.; Dehm, G.; Raabe, D.; Li, Z.: Acta Mater. 185 (2020) 218.
4. Wu, G.; Balachandran, S.; Gault, B.; Xia, W.; Liu, C.; Rao, Z.; Wei, Y.; Liu, S.; Lu, J.; Herbig, M.; Lu, W.; Dehm, G.; Li, Z.; Raabe, D.: Adv. Mater. 32 (2020) 2002619.
5. Wu, G.; Liu, C.; Brognara, A.; Ghidelli, M.; Bao, Y.; Liu, S.; Wu, X.; Xia, W.; Zhao, H.; Rao, J.; Ponge, D.; Devulapalli, V.; Lu, W.; Dehm, G.; Raabe, D.; Li, Z. Mater. Today 51 (2021) 6.
6. Liu, C.; Li, Z.; Lu, W.; Bao, Y.; Xia, W.; Wu, X.; Zhao, H.; Gault, B.; Liu, C.; Herbig, M.; Fischer, A.; Dehm, G.; Wu, G.; Raabe, D.: Nat. Commun. 12 (2021) 5518.
7. Rao, Z; Dutta, B.; Körmann, F.; Ponge, D.; Li, L.; He, J.; Stephenson, L.; Schäfer, L.; Skokov, K.; Gutfleisch, O.; Raabe, D.; Li, Z.: Phys. Rev. Mat. 4 (2020) 014402.
8. Rao, Z; Dutta, B.; Körmann, F.; Lu, W.; Zhou, X.; Liu, C.; da Silva, A.; Wiedwald, U.; Spasova, M.; Farle, M.; Ponge, D.; Gault; B. Neugebauer, J.; Raabe, D.; Li, Z.: Adv. Funct. Mat. 31 (2021) 2007668.

Developing high-strength and damage-tolerant crystalline thin-film materials

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Wear-related energy loss and component damage, including friction and remanufacturing of components that failed by surface contacts, has an incredible cost. While high-strength materials generally have low wear rates, homogeneous deformation behaviour and the accommodation of plastic strain without cracking or localised brittle fracture are also crucial for wear-resistant metals. High-strength and tough coatings have a large role to play within this space, and in this interdepartmental project we have developed both techniques such as high-throughput tensile testing [1], and advanced materials systems such as MAX phase Cr_2AlC coatings [2] and novel interstitial solid solution strengthened thin films [3], to address this challenge.

Amorphous-crystalline nanocomposite thin-films have also been found to promote an ultrahigh yield strength with large deformability, as shown for the Fe-Co-Cr-Ni-Si-B system [4]. We also present a new strategy to develop thermally stable, high-strength and deformable crystal-glass nanocomposites (Fig. 1a-d) through a thermodynamically guided alloy design approach, which mimics the mutual stabilisation principle known from symbiotic ecosystems. We realised this in form of a model Cr-Co-Ni (crystalline, ~18 nm-thick)/Ti-Zr-Nb-Hf-Cr-Co-Ni (amorphous, ~12 nm-thick) laminate composite alloy [5]. The symbiotic alloy has an ultrahigh compressive yield strength of 3.6 GPa and large homogeneous deformation of ~15 % strain at ambient temperature, with ~200 K higher crystallisation temperature compared to the original TiZrNbHf-based amorphous phase. The elemental partitioning among adjacent amorphous and crystalline phases leads to their mutual thermodynamic and mechanical stabilisation, opening up a new symbiotic approach for stable, strong and ductile materials.

These design concepts were then adapted to develop wear-resistant metals, which achieve their superior properties via *in situ* formation of a strong and deformable oxide nanocomposite surface during wear in an oxidative environment. This was realised for a multi-component $(TiNbZr)_{75}Ag_{25}$ alloy, by blending a TiNbZr medium-entropy alloy with Ag [6]. A ball-on-disk sliding test at maximum contact stress of 1.0 GPa revealed an alloy with low friction coefficient in air. From this tribological contact, a ~400 nm thick nanocomposite layer is formed on the crystalline alloy surface, with ~10 nm-sized Ag nanocrystals embedded in the amorphous oxide matrix (Fig. 1e). Nanopillar compression tests of the friction-induced nanocomposite reveal a yield strength of 2.4 GPa and homogeneous deformation to 20 % strain [6]. This strong and deformable oxide nanocomposite surface promotes an ultralow wear rate of the $(TiNbZr)_{75}Ag_{25}$ alloy, compared to TiNbZr or $(TiNbZr)_{75}Ag_{25}$ alloy tested in Ar atmosphere, demonstrating a pathway for designing ultra-wear resistant alloys.

Thermal stability and oxidation resistance are also defining properties for advanced material systems, including coated components used in e.g. energy conversion applications. The MAB phase MoAlB is a promising candidate for such applications since the formation of a dense and adherent alumina scale has been reported for bulk samples. X-ray phase pure, orthorhombic MoAlB coatings were grown at 700 °C for the first time [7]. These coatings exhibit similar oxidation behaviour compared to bulk MoAlB since the oxide scale formed on MoAlB coatings after oxidation at 1200 °C for 30 min was similar to the one extrapolated for bulk MoAlB (Fig. 2), with improved oxidation kinetics than bulk Ti_2AlC. High-resolution transmission electron microscopy reveals an interplanar spacing which is in excellent agreement with the lattice parameter determined by X-ray diffraction and *ab initio* predictions [7] as well as literature reports. In selected area electron diffraction and compositional area fraction analysis, additional, yet unidentified, Al-rich and O-rich minority phases were observed [8]. These findings are significant because the formation of 2D MBenes (2D metal borides) was observed in the vicinity of the O-rich segregations [8]. In addition to exploring the direct synthesis of 2D MBenes by magnetron sputtering, current research focuses on improving the crystal quality [9] and purity of the MoAlB coatings and heterostructures (see p. 26).

References

1. Völker, B.; Du, C.; Fager, H.; Rueß, H.; Soler, R.; Kirchlechner, C.; Dehm, G.; Schneider, J.M.: Surf. Coat. Tech. 390 (2020) 125645.
2. Völker, B.; Stelzer, B.; Mráz, S.; Rueß, H.; Sahu, R.; Kirchlechner, C.; Dehm, G.; Schneider, J.M.: Mater. Des. 206 (2021) 109757.
3. Liu, C; Lu, W.; Xia, W.; Du, C.; Rao, Z.; Best, J.P.; Brinckmann, S.; Lu, J.; Gault, B.; Dehm, G.; Wu, G.; Li, Z.; Raabe, D.: Nature Comm. accepted.
4. Wu, G.; Balachandran, S.; Gault, B.; Xia, W.; Liu, C.; Rao, Z.; Wei, Y.; Liu, S.; Lu, J.; Herbig, M.; Lu, W.; Dehm, G.; Li, Z.; Raabe, D.: Adv. Mater. 32 (2020) 2002619.
5. Wu, G.; Liu, C.; Brognara, A.; Ghidelli, M.; Bao, Y.; Liu, S.; Wu, X.; Xia, W.; Zhao, H.; Rao, J.; Ponge, D.; Devulapalli, V.; Lu, W.; Dehm, G.; Raabe, D.; Li, Z. Mater. Today 51 (2021) 6.
6. Liu, C.; Li, Z.; Lu, W.; Bao, Y.; Xia, W.; Wu, X.; Zhao, H.; Gault, B.; Liu, C.; Herbig, M.; Fischer, A.; Dehm, G.; Wu, G.; Raabe, D.: Nat. Commun. 12 (2021) 5518.

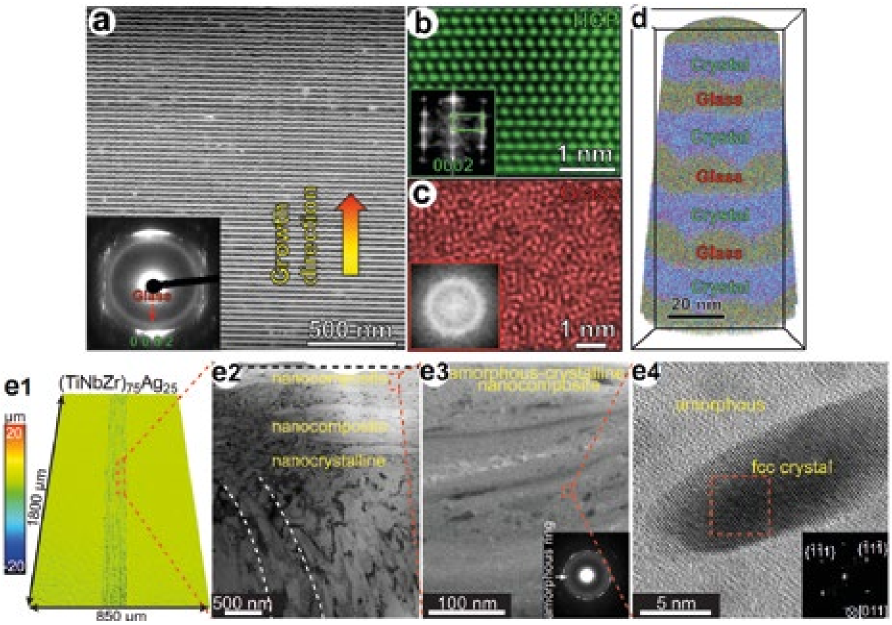

Fig. 1: *Microstructure and composition of the symbiotic alloy. High angle annular dark-field scanning transmission electron microscopy (STEM) image (a) with selected area electron diffraction (SAED) pattern (inset) shows an amorphous ring and diffraction pattern with a strong {0 0 0 2} texture. Side-view high-resolution STEM images showing hcp crystalline CrCoNi phase probed along the <1 1 -2 0> zone axis (b) and maze-like pattern of the amorphous phase (c). The corresponding fast Fourier transformation (FFT) insets show that the {0 0 0 2} plane is perpendicular to the growth direction and the glass phase presents a typical diffuse ring feature. 3D reconstruction of an atom probe tomography (APT) dataset, showing nano-laminated structure (d). 3D profile of* $(TiNbZr)_{75}Ag_{25}$ *wear surface is shown (e1) together with: TEM image presenting a ~400-nm-thick amorphous-crystalline nanocomposite on the surface (e2); Annular bright field (ABF)-STEM image displaying the structure of the nanocomposite (e3, inset SAED); Atom-resolved ABF-STEM image highlighting a fcc structured nanocrystal embedded in the amorphous matrix (e4, inset FFT).*

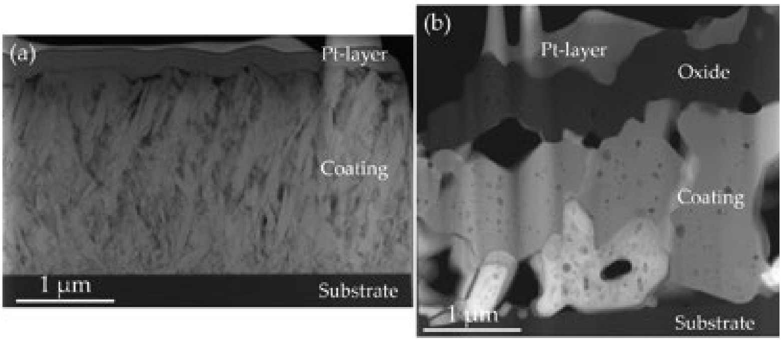

Fig. 2: *STEM images of close-to-stoichiometric MoAlB coating; (a) as-deposited, (b) after oxidation in ambient air at 1200 °C. The top layer corresponds to the Pt protection layer, which is deposited during the focussed ion beam (FIB) sample preparation, adapted from [7].*

7. Achenbach, J.-O.; Sahu, R.; Völker, B.; Hans, M.; Primetzhofer, D.; Miljanovic, D.J.; Scheu, C.; Schneider, J.M.: Coatings 9 (2019) 510.

8. Sahu, R.; Bogdanovski, D.; Achenbach, J.-O.; Zhang, S.; Hans, M.; Primetzhofer, D.; Schneider, J. M.; Scheu, C.: Nanoscale 13 (2021) 18077.

9. Evertz, S.; Pöllmann, P.; Holzapfel, D.M.; Mayer, E.; Schneider, J.M.: J. Eur. Ceram. Soc. 41 (2021) 6302.

Design of tough metallic glasses

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The promising mechanical properties of metallic glasses (MG) such as high hardness, yield strength, and toughness [1] are desirable to exploit for structural applications. Monolithic MGs lack grains and grain boundaries; thus, the mechanical properties of MGs are depending on the chemistry as well as processing and testing conditions. However, despite the promising properties, catastrophic failure is often observed, especially under uniaxial tension.

While most investigations on the mechanical properties of MGs have focussed on the topology of the atomic motifs, attention must also be placed on the nature of the local chemical bonding, which is the basis of atomic cohesion and hence mechanical properties [2]. However, while elastic properties can be directly calculated from *ab initio* calculations, mechanical properties such as fracture toughness, which involves the rearrangement of atoms, need new predictors and their validation [2]. Here, we present a predictor for the fracture toughness of MGs based on the electronic structure.

The brittle to tough transition cannot be universally predicted based on the Poisson's ratio [2]. Instead, based on a comparison of the *ab initio* electronic structure and the fracture toughness of $Cu_{70}Zr_{30}$ and $Pd_{57}Al_{24}Y_8Cu_{11}$, we hypothesize that the fraction of hybridized bonds with respect to the overall bonds is a qualitative fingerprint of fracture toughness [3]: The lower the fraction of hybridized bonds, the higher the fracture toughness. Hence, the fraction of metallic bonds needs to be maximized to enhance fracture toughness [4]. Since the fraction of hybridized bonds can be obtained from *ab initio* calculations by calculating the crystal orbital overlap population (COOP) [5], the predictor for fracture toughness focuses on minimizing the COOP. For purely metallic bonding the electrons at the Fermi level E_f are delocalized. Hence, for an ideal MG in terms of toughness, COOP(E_f) = 0 [2, 4].

The experimental fracture toughness of MG scales with the COOP(E_f) (Fig. 1) [4]. Based on the fit of the data, the unknown fracture toughness of a $Pd_{57}Al_{24}Y_8Ni_{11}$ can be predicted to be 95 ± 20 MPa $m^{0.5}$ [4]. However, as indicated by the negative COOP(E_f) in Fig. 1a, not only the fraction of hybridized bonds is crucial, but also the anti-bonding bonding character at E_f, which leads to a higher energy in the system and promotes bond separation [4].

To critically appraise this new predictor for the fracture toughness of MGs, micro-mechanical bending tests on magnetron sputtered $Pd_{58}Al_{25}Y_5Ni_{12}$ thin films were performed. In these experiments, the cantilevers showed formation of multiple shear bands confined by the stress gradient (Fig. 1b), with no crack extension [4]. Hence, the fracture toughness exceeds the boundaries of micromechanical fracture experiments, which is consistent with the high toughness predicted for this material [4]. However, despite the large plasticity observed here in a complex stress state and with a small size, strain localization in uniaxial tension can still lead to catastrophic failure by strain localization.

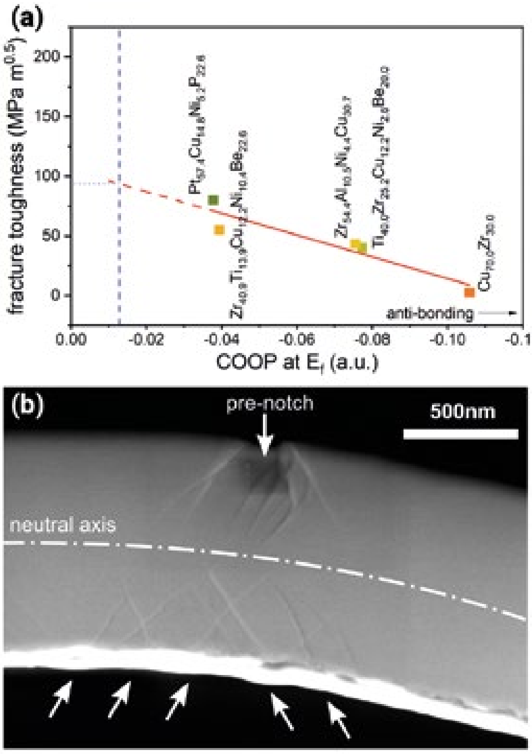

Fig. 1: *(a) Fracture toughness of five different MGs as a function of crystal orbital overlap population (COOP) at the Fermi level E_f. The red solid line is a linear fit of the data points. The dashed blue line indicates the COOP(E_f) of $Pd_{57}Al_{24}Y_8Ni_{11}$ and the dotted blue line indicates the predicted fracture toughness. (b) In-lens-SE micrograph of the notch region of a micro-cantilever after micro-bending to 4 µm deflection. Severe shear band formation is observed around the notch as well as on the compressive side of the cantilever (see arrows) [4].*

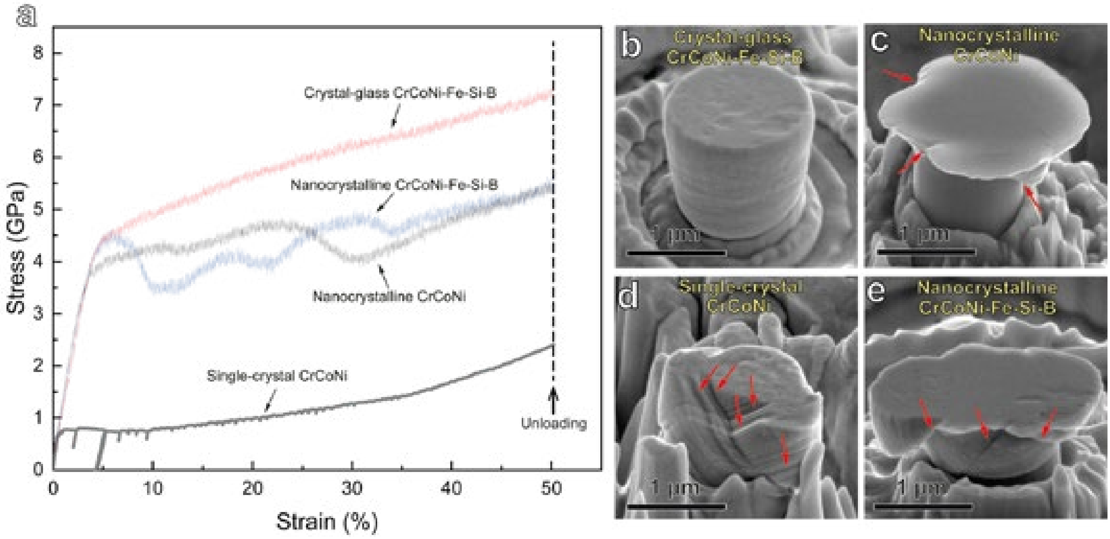

Fig. 2: *Mechanical properties of a CrCoNi-Fe-Si-B crystal–glass high-entropy nanocomposite. (a) Compressive engineering stress–strain curves of the crystal-glass CrCoNi-Fe-Si-B high-entropy nanocomposite, nanocrystalline CrCoNi-Fe-Si-B, nanocrystalline CrCoNi, and single-crystal CrCoNi pillar samples, each with the same diameter of 1 µm. (b)–(e) Scanning electron microscopy (SEM) images of the same samples after compression to an identical engineering strain of 50 %. Some of the shear bands in (c) and (e) and slip bands in (d) are indicated by red arrows.*

Glass-matrix composite materials can prevent strain localization by exploiting plasticity of MGs for spatially confined geometries. By combining MGs with e.g. nanocrystalline high-entropy alloys (HEAs), ductile materials with near-theoretical strength can be obtained [6].

By doping a magnetron sputtered Cr-Fe-Co-Ni HEA with the glass-forming elements B and Si, a nanocomposite structure composed of a ~1 nm-thick amorphous phase surrounding the 8 nm-thick crystalline HEA columnar grains was formed [6]. In micro-mechanical compression tests, the crystal-glass composite exhibits a yield strength of 4.1 GPa (Fig. 2), which is higher than that of the nanocrystalline reference alloy with a similar composition. Moreover, the crystal-glass high-entropy nanocomposite reveals homogeneous plastic deformation without formation of visible shear bands (Fig. 2) [6].

During plastic deformation of the crystal-glass nanocomposite, the nanograins are gradually subdivided, creating additional interfaces against dislocation motion [6]. The formation of additional interfaces compensates the work softening from de-twinning. Stress-concentrations by dislocation pile-ups at the grain boundaries are prevented by the absorption of dislocations in the amorphous phase. The interplay of continuous partial dislocation formation, movement, and annihilation promotes the homogeneous and compatible plastic deformation of the crystal-glass composite [6].

In conclusion, the here introduced predictor for fracture toughness based on the electronic structure obtained by *ab initio* calculations enables the identification of MG compositions with promising fracture toughness *in silico*. This offers a resource- and time-efficient navigation through the vast composition space of MGs. Combining the beneficial properties - high yield strength and toughness – with the high ductility of crystals, a new material class called crystal-glass high-entropy nanocomposite was developed. It reveals enhanced yield strength and ductility compared to the fully crystalline reference alloys. With both the prediction of fracture toughness and tuneable yield strength in these crystal-glass composites, a toolbox for tailoring mechanical properties of crystal-glass HEA composites has become available.

References

1. Ashby, M.; Greer, A. L.: Scr. Mater. 54 (2006) 321.
2. Evertz, S.; Schnabel, V.; Köhler, M.; Kirchlechner, I.; Kontis, P.; Chen, Y.-T.; Soler, R.; Nagamani Jaya, B.; Kirchlechner, C.; Music, D.; Gault, B.; Schneider, J. M.; Raabe, D.; Dehm, G.: Front. Mater. 7 (2020) 89.
3. Schnabel, V.; Nagamani Jaya, B.; Köhler, M.; Music, D.; Kirchlechner, C., Dehm, G.; Raabe, D.; Schneider, J. M.: Sci. Rep. 6 (2016) 36556.
4. Evertz, S.; Kirchlechner, I.; Soler, R.; Kirchlechner; C.; Kontis, P.; Bednarcik, J.; Gault, B.; Dehm, G.; Raabe, D.; Schneider, J. M.: Mater. Des. 186 (2020) 108327.
5. Hoffmann, R.: Angew. Chem. Int. Edit. 26 (1987) 846.
6. Wu, G.; Balachandran, S.; Gault, B.; Xia, W.; Liu, C.; Rao, Z.; Wei, Y.; Liu, S.; Lu, J.; Herbig, M.; Lu, W.; Dehm, G.; Li, Z.; Raabe, D.: Adv. Mater. 32 (2020) 202619.

Software development

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Recent developments in experimental techniques and computer simulations provided the basis to achieve many of the breakthroughs in understanding materials down to the atomic scale. While extremely powerful, these techniques produce more and more complex data, forcing all departments to develop advanced data management and analysis tools as well as investing into software engineering expertise. As a consequence, a MPIE software task force has been established (see p. 70), where software engineering and simulation software expertise developed e.g. in the CM department is utilized. Here, we have selected a few examples to demonstrate how these developments help to propel science both in experiment and theory.

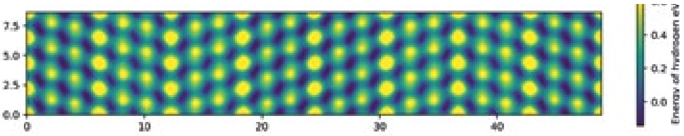

Fig. 1: *Free energy surface of H next to a Σ5 grain boundary in fcc Ni. The result has been achieved by developing a novel metadynamics schema with periodic collective coordinate within regular hackathons on hydrogen diffusion.*

An integrated development environment

On p. 72, pyiron has been introduced as an integrated platform for materials simulations and data management. At the same time, pyiron has also modified the culture of research in the CM department towards a stronger focus on software development. For the outer community, this is visible by a growing number of journal publications that are combined with executable code required for the physical analysis shown in the paper [1]. This code is provided within binder containers, such that the user can directly employ the simulations on cloud resources without the need of cumbersome installations on the local hardware. One example of this kind is the fully automated approach to calculate the melting temperature of elemental crystals [2]. The manuscript explains many of the technical challenges connected to the interface method, used for melting point calculations. The underlying complex simulation protocols are provided as pyiron notebooks, which allows a much more interactive access, reproduction and exploration of the underlying concepts than a written manuscript.

More significant for the research inside the CM department is, however, the cooperative development of new physical concepts in regular coding sessions. Several teams are currently meeting for several hours on a weekly basis, in order to test new physical ideas by directly implanting and executing them in pyiron notebooks. The physically intuitive structure of the pyiron environment allows such a research approach, without spending time on many command lines, taxonomy and syntax. For example, one group is exploring the physical nature of thermo desorption spectra (TDS) for hydrogen in metals. This involves the automatic detection of favourable sites for H incorporation into complex microstructure features (such as grain boundaries, Fig. 1), the analysis of possible jump processes between these sites and the evaluation of the H dynamics by kinetic Monte-Carlo simulations. Another group is connecting the workflows in pyiron notebooks with the semantic description of materials knowledge in ontologies, opening new routes of software development based on physical concepts rather than command lines.

Machine learning for solid mechanics

In the MA department, an artificial intelligence (AI)-based solution for predicting the stress field in inhomogeneous elasto-plastic materials was recently developed [3]. The training data for a convolutional neural network (CNN) were generated using DAMASK (Düsseldorf Advanced Material Simulation Kit) for modelling multi-physics crystal plasticity (see p. 73). Both, cases of materials with elastic and elasto-plastic behaviour, are considered in the training data. The trained CNN model is able to predict the von Mises stress fields (Fig. 2) orders of magnitude faster than conventional solvers. In the case of elasto-plastic material behaviour, conventional solvers rely on an iterative solution scheme for the nonlinear problem. The AI-based solver is able to predict the correct solution in one step forward evaluating of the CNN. This significantly reduces the implementation complexity and computation time. The augmentation of such a fast stress estimator with finite element-based solvers will open new avenues for the multi-scale simulation of materials.

Software tools for atom probe microscopists

In order to achieve scientific conclusions from atom probe tomography (APT), these data have to be processed into tomographic reconstructions, i.e. 3D models that specify the approximate original locations of the atoms in the field-evaporated specimen. Representing point clouds, these models are post-processed with methods from the field of spatial statistics, computational geometry, and AI resulting in quantified (micro)structural features or patterns that can be related to descriptors in physics-based models at the continuum scale such as dislocations, grain and phase boundaries, or precipitates.

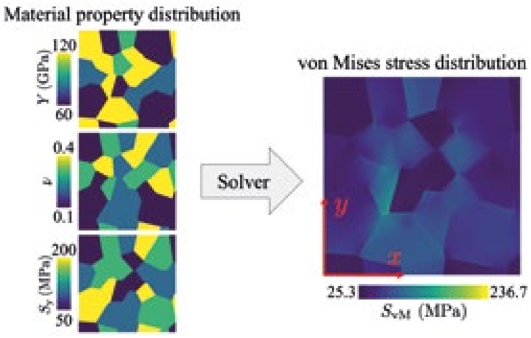

Fig. 2: *A typical mechanical solver relating the input material property distribution to an output von Mises stress distribution. For specific cases, the solver could be replaced by a convolutional neural network, which is orders of magnitude faster than the solver.*

It is important to quantify the eventual uncertainty and sensitivity of the descriptors as a function of the parameterization. To this end, though, the employed proprietary tools face substantial practical and methodological limitations with respect to the accessibility of intermediate (numerical and geometry) data and metadata they offer, the transparency of the algorithms, or the functionalities they provide to script and interface proprietary tools with open-source software of the atom probe community.

Involving APT domain specialists from the MA and computational materials scientists from the CM department, we implemented a toolbox of complementary open-source software for APT, which makes efficient algorithms of the spatial statistics, computational geometry, and crystallography community accessible to support scientists with quantifying the parameter sensitivity of APT-specific methods they use. Our tools exemplify how open-source software enables to build tools with programmatically customizable automation and high-throughput characterization capabilities, whose performance can be augmented by cluster computing. We show how to automate and make exactly reproducible the execution of microstructural feature characterization [4 - 6] for atom probe data.

Analysis tools for complex imaging and spectroscopy

One example of software development in the SN department is the creation of analysis workflows for data from transmission electron microscopes (TEM). Typically, most experimentalists use graphical user interface (GUI) based tools to process their data. Many of these tools are proprietary and licensed by the hardware manufacturer. The analysis options in this software are limited and rarely reproducible. In addition, with the advancement in detector technology and other instrumentation, the size and complexity of data is ever increasing. This calls for highly customizable analysis tools. This is also necessary for bridging the gap between the powerful machine learning and artificial intelligence libraries in the scientific python ecosystem and the workflow of the average experimentalist.

The experimental TEMMETA [7] library was built to address the most common analysis needs offered in software like ImageJ or Digital Micrograph, such as drawing line profiles, selecting subsets of the data, visualizing the data, and calculating strain fields from atomically resolved images. TEMMETA also has the feature of automatically storing information about each operation performed on data in a history attribute, thereby making it easier to reproduce the analysis. As development progressed, it became clear that the TEMMETA project had many of the same aims as the Hyperspy project, a library that has existed for a longer time and already has an established community of users and developers. Therefore, it was decided to integrate the novelties of the TEMMETA project into Hyperspy [8].

File parsers to a common representation are essential for bringing the full functionality of Hyperspy to any kind of microscopy data. The SN department is increasingly adopt-ing 4D-STEM as an advanced TEM based technique. It produces large and rich datasets of diffraction patterns collected on a grid of scan points. A typical workflow for these datasets relevant to metallurgists is analysing local crystal orientation via template matching. Existing proprietary software solutions could not handle the data from pixelated detectors, hence a custom built fast and scalable solution [9] was implemented in a package that builds on Hyperspy. This should make the transparent analysis of 4D-STEM datasets more accessible to researchers at the MPIE and all over the world.

References

1. https://pyiron.org/publications/
2. Zhu, L.-F.; Janssen, J.; Ishibashi, S.; Körmann, F.; Grabowski, B.; Neugebauer, J.: Comp. Mater. Sci. 187 (2021) 110065.
3. Mianroodi, J. R., H. Siboni, N. & Raabe, D.: npj Comput. Mater. 7 (2021) 99.
4. Kühbach, M.; Bajaj, P.; Zhao, H.; Celik, M.H.; Jägle, E.A.; Gault, B.: npj Comput. Mat. 7 (2021) 21.
5. Kühbach, M.; Kasemer, M.; Gault, B.; Breen, A.: J. Appl. Cryst. 54 (2021) 1490.
6. Kühbach, M.; London, A.J.; Wang, J.; Schreiber, D.K.; Mendez, M.F.; Ghamarian, I.; Bilal, H.; Ceguerra, A.V.: Microsc. Microanal. (2021). doi: 10.1017/S1431927621012241.
7. Cautaerts, N.; Janssen, J.: Zenodo: din14970/TEMMETA v0.0.6 (2021). doi.org/10.5281/zenodo.5205636.
8. de la Peña, F.; Prestat, E.; Fauske, V.T.; Burdet, P.; Furnival, T.; Jokubauskas, P.; Lähnemann, J.; Nord, M.; Ostasevicius, T.; MacArthur, K.E.; Johnstone, D.N.; Sarahan, M.; Taillon, J.; Aarholt, T.; pquinn-dls; Migunov, V.; Eljarrat, A.; Caron, J.; Poon, T.; Mazzucco, S.; Martineau, B.; actions-user; Somnath, S.; Slater, T.; Francis, C.; Tappy, N.; Walls, M.; Cautaerts, N.; Winkler, F.; Donval, G.: Zenodo: hyperspy v1.6.4 (2021). doi.org/10.5281/zenodo.5082777.
9. Cautaerts, N.; Crout, P.; Ånes, H. W.; Prestat, E.; Jeong, J.; Dehm, G.; Liebscher, C.H.: cond-mat arXiv:2111.07347

Integrated workflows for materials and data science

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[1]CM, [2]MA

Integrated Computational Materials Engineering (ICME) is one of the emerging hot topics in Computational Materials Simulation during the last years. It aims at the integration of simulation tools at different length scales and along the processing chain to predict and optimize final component properties.

The realization of ICME concepts necessitates a workflow system, which on the one hand allows a flexible combination of different simulation tools and on the other hand enables the data exchange between these different tools. In addition, the workflow system should follow the FAIR (Findable, Accessible, Interoperable, and Re-usable) principle for sustainable materials development and offer a generic user interface to handle the different simulation tools.

pyiron (see p. 72, [1]) developed by the CM department is such a workflow platform. Originally designed for the integrated development and management of workflows on the atomistic scale, pyiron has been systematically extended during the last years to serve as a general workflow platform. In this process pyiron has been modularized. Besides the core module (pyiron_base) there exist three additional modules. pyiron_atomistic offers the original functionality on the atomistic scale; pyiron_continuum integrates continuum scale simulation tools like e.g. DAMASK (see p. 73, [2]) developed in the MA department; pyiron_experimental finally allows handling experimental data. The last module, which currently focuses on (scanning) transmission electron microscopy ((S)TEM) data, extends the application of ideas originally developed in computational materials simulation to experimental data and thus allows the creation of mixed experimental and simulation workflows following the FAIR principle.

In the early times of ICME coupling of different simulation tools was usually achieved on a one-to-one basis. This means the output of program 1 was converted to serve as input for program 2. One of the goals of the Federal Ministry of Education and Research (BMBF) initiative 'Material*Digital*' (see p. 80) is to overcome this direct coupling using ontology-based concepts. Here ontologies serve as an abstraction layer to define relations between data and concepts, which can be exploited for workflows and program interfaces. Such abstraction allows an exchange of different simulation tools that serve for a given workflow the same purpose. In this way workflows can be defined independently of the underlying simulation tools. Following a BMBF call in the framework of 'Material*Digital*', several academic projects have been initiated to define such workflows. The MPIE participates in the project 'Stahl*Digital*´ together with the Fraunhofer Institute for Mechanics of Materials in Freiburg and the Institute for Applied Informatics (InfAI) at the University of Leipzig. The aim of 'Stahl*Digital*´ is to define different workflows along the production chain and for the usage of steel sheets. These workflows will be based on ontologies, which describe both the material (two different steels) and the process chain (from hot rolling to crash performance) (Fig. 1).

Two different pre-existing workflow platforms will be extended for this purpose, namely SymPhoNy [3] developed by the Fraunhofer IWM and pyiron developed by the MPIE (Fig. 2). While SymPhoNy already allows the usage of ontologies for the workflow description, pyiron is currently extended accordingly.

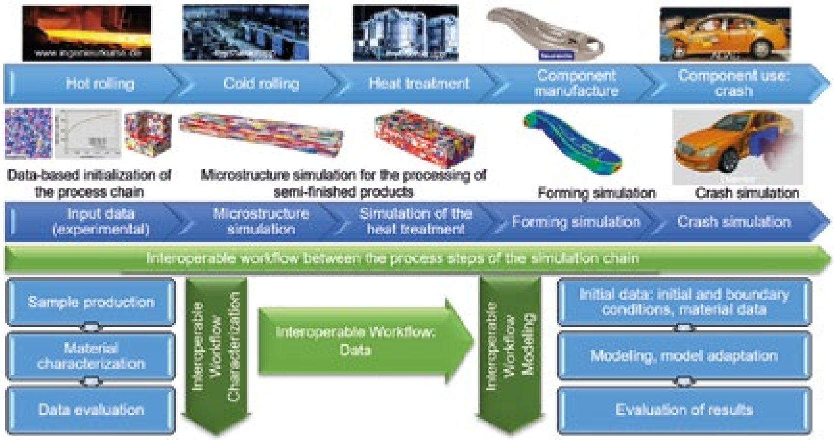


Fig. 1: *Workflows within the project 'StahlDigital'.*

In the following, we showcase two prototypical workflows as they can be easily defined and executed using pyiron:

1. A simulation workflow combining atomic scale simulation with continuum scale simulation.
2. A data-mining workflow evaluating simulation results.

pyiron workflows are developed as Jupyter notebooks [4], which ensures full reproducibility. In addi-

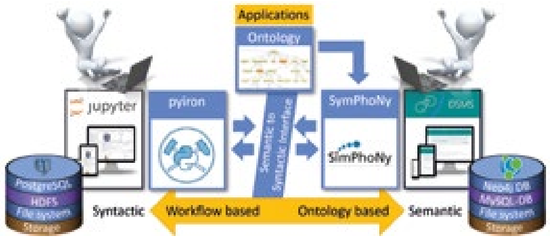


Fig. 2: *Workflow platforms used in 'StahlDigital'.*

tion, on top of the pyiron functionality any Python library can be easily included to perform special tasks.

Polycrystal elastic properties

Full field DAMASK simulations are well suited for the numerical homogenization of single crystal properties. However, often these single crystal properties are not known a priori. Here we are interested in the elastic properties of aluminium with different additions of magnesium. While the elastic constants of the pure materials are readily available in the literature, this is not true for the alloys. However, good interatomic potentials are available for the Al-Mg system. So, we can perform molecular dynamics simulations to determine the single crystal elastic constants. These are then passed into DAMASK, which is used to probe the elastic behaviour of a polycrystal under different loading conditions.

The full workflow consists of the following steps:

1. Minimize the structure for a pure Al cubic cell
2. Create special quasirandom structures (SQS) for different Mg concentrations
3. Select the interatomic potential
4. Run LAMMPS [5] simulations to determine elastic constants for each Mg concentration
5. Fit elastic constants as function of Mg concentrations (Fig. 3)
6. Create polycrystal volume element for DAMASK
7. Define loadcases for DAMASK
8. Inject elastic constants into DAMASK material file
9. Run DAMASK simulations
10. Determine homogenized elastic constants

Assessment of empirical potentials for fcc-Al

The idea behind this workflow is to compare various empirical potentials with respect to their performance for ground state properties of fcc-Al. The workflow consists of the following steps:

1. Get the list of available LAMMPS potentials
2. Create a Murnaghan job for each potential
3. Collect results in a table (Tab. 1)

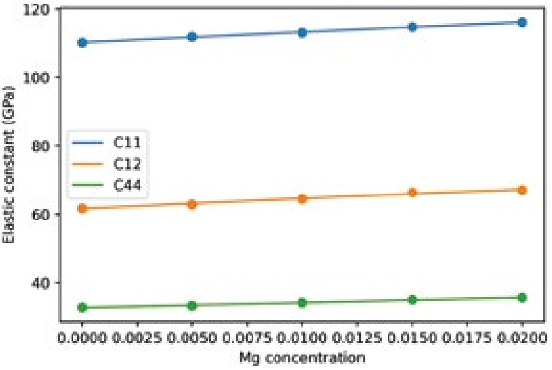
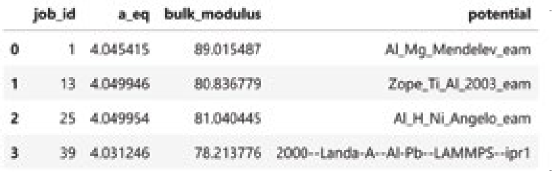


Fig. 3: *Single crystal elastic constants of an Al-Mg solid solution as function of Mg concentration.*

While both workflows seem to be not very complex, they potentially need to run a huge number of individual simulation jobs. In this case pyiron takes full care of distributing the individual jobs to computing nodes, collecting the results, and at the same time record important meta data about the jobs in a SQL data base.



	job_id	a_eq	bulk_modulus	potential
0	1	4.045415	89.015487	Al_Mg_Mendelev_eam
1	13	4.049946	80.836779	Zope_Ti_Al_2003_eam
2	25	4.049954	81.040445	Al_H_Ni_Angelo_eam
3	39	4.031246	78.213776	2000--Landa-A--Al-Pb--LAMMPS--ipr1

Tab. 1: *Screenshot of a table containing equilibrium lattice constant (a_eq) and bulk modulus of Al for different potentials.*

In summary, workflow management platforms like pyiron allow easy definition and execution of complex simulation projects following the FAIR principle. The connection to ontologies will make them even more flexible and easy to use.

References

1. Janssen, J.; Surendralal, S.; Lysogorskiy, Y.; Todorova, M.; Hickel, T.; Drautz, R.; Neugebauer, J.: Comput. Mater. Sci. 163 (2019) 24.
2. Roters, F.; Diehl, M.; Shanthraj, P.; Eisenlohr, P.; Reuber, C.; Wong, S.L.; Maiti, T.; Ebrahimi, A.; Hochrainer, T.; Fabritius, H.-O.; Nikolov, S.; Friák, M.; Fujitai, N.; Grilli, N.; Janssens, K.G.F.; Jia, N.; Kok, P.J.J.; Ma, D.; Meier, F.; Werner, E.; Stricker, M.; Weygand, D.; Raabe, D.: Comput. Mater. Sci. 158 (2019) 420.
3. https://www.simphony-project.eu/
4. https://jupyter.org/
5. Thompson, A. P.; Aktulga, H. M.; Berger, R.; Bolintineanu, D. S.; Brown, W. M.; Crozier, P. S.; in 't Veld, P. J.; Kohlmeyer, A.; Moore, S. G.; Nguyen, T. D.; Shan, V.; Stevens, M. J.; Tranchida, J.; Trott, C.; Plimpton, S. J.: Comp. Phys. Comm. 271 (2022) 10817.

Advancing atom probe towards true atomic-scale analytical tomography

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Atom probe tomography (APT) is one of the MPIE's key experiments for understanding the interplay of chemical composition in very complex microstructures down to the level of individual atoms. In APT, a needle-shaped specimen (tip diameter ≈100nm) is prepared from the material of interest and subjected to a high voltage. Additional voltage or laser pulses trigger the evaporation of single ions from the tip. Accelerated by the voltage, the ions fly towards a position-sensitive channel-plate detector. The time-of-flight reveals the mass-to-charge ratio from which the elemental identity of each detected ion is deduced. Datasets of up to 10^9 ions are routinely acquired. From a data science perspective, APT datasets are a gold mine. APT data sets are huge, noisy, and full of more or less hidden patterns.

Data evaluation involves first building a virtual 3D point cloud in which the position of each ion prior to field evaporation is reconstructed. Beyond this first step, unbiased, quantitative analysis of the information encoded in the data remains challenging. A few examples: (1) elemental identities must be correctly assigned from a potentially complex mass spectrum that contains not only atomic ions, but also often metastable small molecular units; (2) the reconstructed positions are affected by uncertainties that blur out any underlying crystalline structure; (3) microstructural features must be extracted – what phases and crystal grains do exist? Where are their boundaries? How, where, and why do compositions change?

To standardize mass spectra analysis, in particular when it comes to identify the most likely ion when multiple candidates exist for the same mass-to-charge ratio, we demonstrated that deep neural networks can be trained to disentangle mass spectra [1]. This has the potential to enormously improve the reproducibility of mapping the mass-to-charge ratio to a set of chemical identity, compared to ranges defined by human inspection which could lead to composition significant variations between experts [2].

Current APT reconstructions are based on a geometrical projection algorithm from the detector position onto a spherical cap, which largely ignores the details of the underlying physics. In reality, trajectory aberrations occur due to the local environment prior to evaporation or mesoscopic field variations associated to surface roughness. The order of evaporation is not exclusively dictated by position, but possibly also by chemical identity and bonding environment, and affects the reconstruction process causing limitations of the spatial resolution. In order to quantify these effects, the group of C. Freysoldt has started systematic theoretical investigations of field evaporation mechanisms. In addition to confirming a material-dependent tendency for roll-over at steps on faceted surfaces, they were also able to demonstrate the crossover from a two-step to a one-step mechanism controlled by the field strength [3]. It was further shown that the surface mobility of hydrogen in presence of the field and the tendency for formation of H-containing ions depend primarily on the charge transfer of the adsorbed H.

Another topic of interest is to recover the crystalline lattice structure even when the positional uncertainty of the reconstruction prevents direct identification. The crystal orientation often gives rise to a systematic intensity pattern on the detector, which can be analysed by deep-learning [4]. Grain boundaries lead to a discontinuous pattern that can be identified and tracked automatically as the data set is sliced along the evaporation order, i.e. a proxy for the depth [5]. This approach allows for direct data filtering and quantification of chemical segregation to the boundary.

Crystallographic information of APT specimen is directly accessible by using field ion microscopy (FIM). FIM is a precursor technique in which an imaging gas is continuously ionized above the surface held below evaporation threshold. A new experimental approach was initiated at the MPIE in 2018 combining FIM with enabled mass spectrometry enabled by a correlative data analysis. We used this so-called analytical FIM to directly image solute segregation to dislocations [6] and to vacancies in Ni-based binary alloys [7]. These experimental investigations were paralleled by theoretical investigations to explain the chemical contrast observed in FIM by an electronic-structure effect.

At larger scales, the formation of phases with different chemical composition is of common interest. When formed at the nanoscale, there can be dozens or even hundreds of small precipitates in a single APT data set. While they are easily visualized by concentration iso-surfaces of key elements, this is impractical to perform quantitative analysis. Therefore, the Paraprobe toolset was developed [6]. It not only allows a scriptable, well-parallelized high-throughput analysis on high-performance computers without live interaction from a human scientist, but also provides standard aggregated (e.g. radial distribution functions) and iso-surface based segmentations in an open data format (based on HDF5) that can be further processed with standard data science and visualization tools.

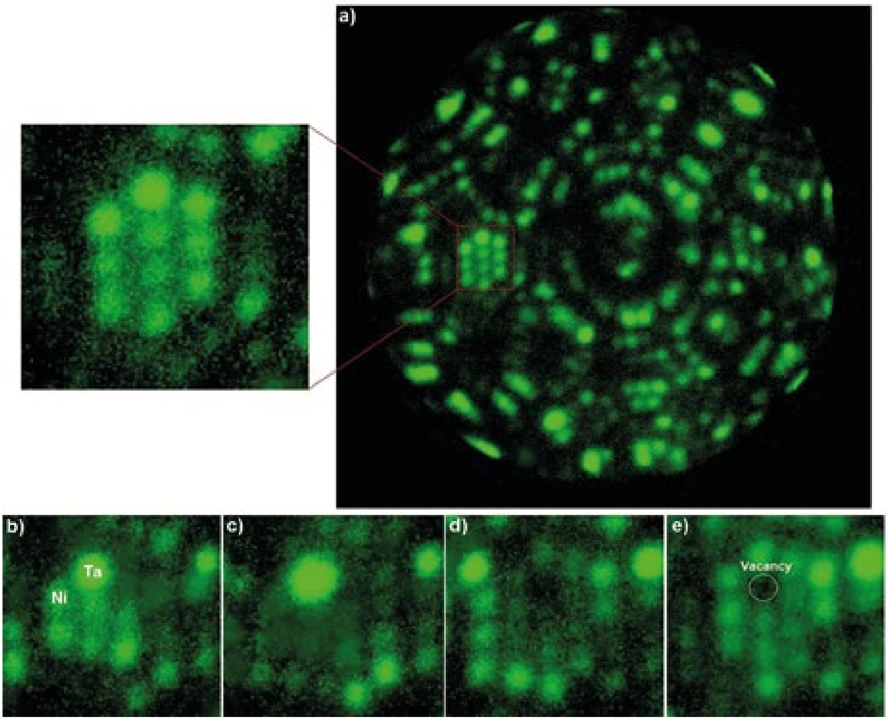

Fig. 1: *Field ion microscopy measurement of Ni-2 at.% Ta alloy. (a) Field ion micrograph zoomed on the (012) planes. (b)-(e) show consecutive atomic layers with identification of Ta, Ni atoms, and vacancy.*

More recently, we have explored alternative 3D phase segmentation algorithms. They start by mapping out the chemical composition of small voxels (10^2-10^3 atoms). Despite the large scatter, clustering in composition space allows to distinguish possible phases without prior information and derive classifiers from it. Next, a DBSCAN cluster analysis in the voxelized dataset labelled according to the composition classifier successfully provides a very fast and robust composition-based segmentation. These segments can then be further analysed based on their geometrical character (e.g. line-like, plate-like, ellipsoidal, etc.) and the application-oriented scientific questions.

References

1. Wei, Y.; Varanasi, R. S.; Schwarz, T.; Gomell, L.; Zhao, H.; Larson, D. J.; Sun, B.; Liu, G.; Chen, H.; Raabe, D.; Gault, B.: Patterns 2 (2021) 100192.
2. Haley, D.; Choi, P.; Raabe, D.: Ultramicroscopy 159 (2015) 338.
3. Ashton, M. W.; Mishra, A.; Neugebauer, J.; Freysoldt, C.: Phys. Rev. Lett. 124 (2020) 176801.
4. Wei, Y.; Gault, B.; Varanasi, R.S.; Raabe, D.; Herbig, N.; Breen, A.J.: Ultramicroscopy 194 (2018) 15.
5. Wei, Y.; Peng, Z.; Kühbach, M.; Breen, A. J.; Legros, M.; Larranaga, M.; Mompiou, F.; Gault, B.: PLoS One 19 (2019) 1.
6. Morgado, F. F.; Katnagallu, S.; Freysoldt, C.; Klaes, B.; Vurpillot, F.; Neugebauer, J.; Raabe, D.; Neumeier, S.; Gault, B.; Stephenson, L.: Scr. Mater. 203 (2021) 114036.
7. Kühbach, M. T.; Bajaj, P.; Zhao, H.; Çelik, M. H.; Jägle, E. A.; Gault, B.: npj Comp. Mat. 7 (2021) 21.

Big data and machine learning in electron microscopy

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Data-rich experiments such as scanning transmission electron microscopy (STEM) provide large amounts of multi-dimensional raw data that encodes, via correlations or hierarchical patterns, much of the underlying materials physics. With modern instrumentation, data generation tends to be faster than human analysis, and the full information content is rarely extracted. We therefore work on automatizing these processes as well as on applying data-centric methods to unravel hidden patterns. At the same time, we aim at exploiting the insights from information extraction to direct the data acquisition to the most relevant aspects, and thereby avoid collecting huge amounts of redundant data.

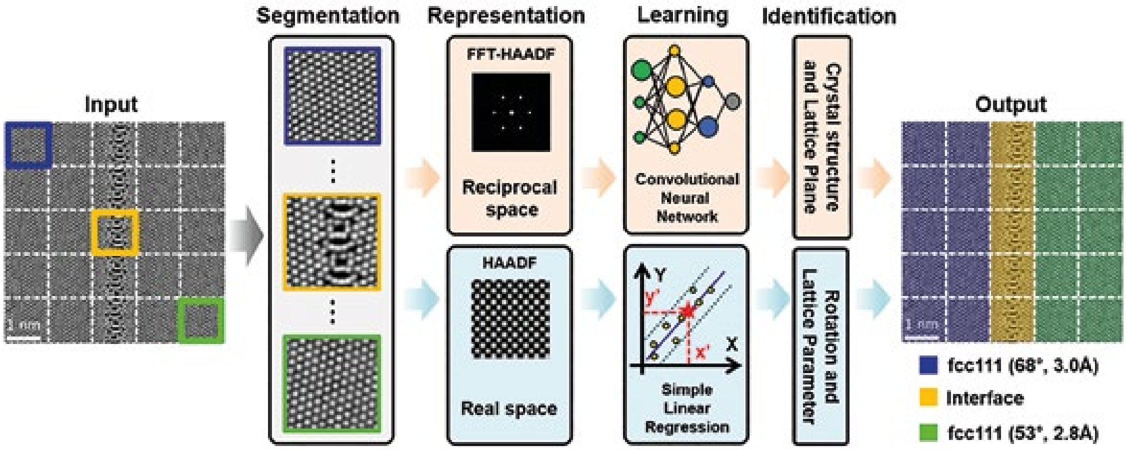

Fig. 1: *Schematic workflow of the classification procedure for experimental STEM images. Here, the input HAADF-STEM image contains two different crystalline regions and one grain boundary. The image is divided into 5 × 5 windows. Three different patches are indicated in the input image: patch (1,1) by the blue, patch (3,3) orange and window (5,5) by the green square. The classification map identifies both blue and green patches as fcc {111}. The orange patches correspond to the grain boundary region, respectively. Image by B.Yeo, L. Ghiringhell, Fritz Haber Institute Berlin.*

The advent of fast electron detectors enabled the collection of full diffraction patterns in each probe-position, while scanning the beam across the electron transparent sample. These 4D-STEM datasets contain rich information on local lattice orientation, symmetry and strain [2]. However, handling and analysing these large-scale datasets, which are typically >10 GB in size requires sophisticated data management infrastructure and algorithms. We are currently developing a flexible and open-source approach within the Python-based pyXem package to index the phase and crystal orientation in scanning nano-beam diffraction datasets [3], which is until now only possible by commercial software. The development will also enable an on-the-fly data analysis in the future by utilizing GPU-accelerated processing.

STEM is also very powerful at revealing the atomic structure even of complex materials. Since under high-angle angular dark field (HAADF) conditions the collected electrons predominantly undergo scattering near the atomic nucleus, the image or atomic column intensity is proportional to their mean atomic number. When aligning the specimen along specific crystallographic directions it is possible to image a 2D projection of the crystal lattice, but also lattice defects such as phase or grain boundaries. Typically, the detection of nanoscale domains and interfaces in complex materials is executed by hand in an iterative manner relying on deep domain knowledge. We have developed a robust symmetry-based segmentation algorithm that solely considers lattice symmetries and hence does not require specific prior insights into the observed structures [4]. It is based on the idea that local symmetries create a self-similarity of the image that can be quantified via Pearson correlation coefficients of image patches. This quantification is very noise-tolerant, in particular if large patches are chosen. By collecting these values for a number of candidate symmetries (100-1000), a characteristic fingerprint of the

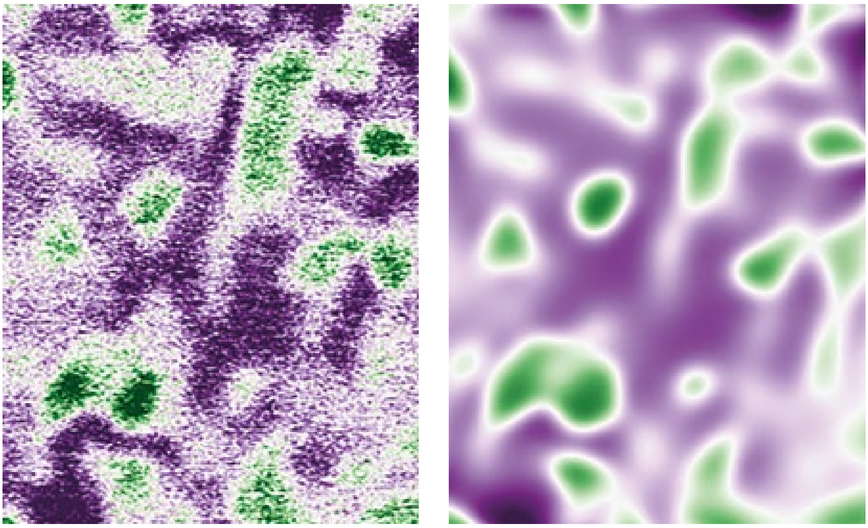

Fig. 2: *Left: Snapshot from in situ STEM-HAADF imaging of the dealloying of a TaTiZrHf high-entropy alloy. Right: snapshot from the phase-field informed neural network representation.*

local crystallographic patterns can be extracted that does not rely on identifying individual atomic columns. These fingerprints can then be taken from various places in the image and allow to filter out recurring patterns by principal component analysis and a subsequent clustering. From this abstract representation, the fingerprints can be classified, yielding a segmentation of the original image without prior training on expected phases. It therefore also works for complex atomic arrangements, e.g., topologically close-packed phases.

To classify and quantify regions autonomously in atomic resolution STEM images, we are currently developing a supervised deep learning approach in a BiGmax collaboration with L. M. Ghiringhelli (Fritz Haber Institute Berlin). The underlying backbone of the classification is a convolutional neural network (CNN), which is trained on simulated STEM images represented by their Fourier transforms. Here, we are considering the basic classes of face-centred cubic (fcc), body-centred cubic (bcc) and hexagonal close packed (hcp) crystal structure for low index orientations. To include uncertainty predictions in the classification, we use Bayesian learning for being able to identify crystal structures or defective regions that the CNN was not trained on. In a second step, the local crystal rotation and lattice parameters are determined by linear regression analysis, which is based on analysing the real space lattice symmetries. In the classification procedure, an experimental image is then subdivided in image patches and the neural network learns which training class is present, its local lattice rotation and lattice constants as shown in Fig. 1. This development is now extended towards a real-space classification procedure also taking into consideration the atomic column intensities to determine local compositional variations and defect structures.

At larger length scales, HAADF allows to monitor phase transformations at elevated temperatures. Such time-resolved measurements yield multiple frames that can be visualized as video sequences. Yet, the quantification of the phase transformation kinetics requires additional processing and simplification of the raw data. In particular, the quantification of intensity gradients is severely hindered by the presence of shot noise. For this, we have explored the use of physics-informed neural networks to map the noisy images to a phase-field model with known analytic structure. The basic idea is to train a smooth machine-learning model to reproduce the measured data, while at the same time requiring it to comply with the phase-field equations, using model parameters that are trained on the fly. It turns out that such an approach is very efficient to obtain a coarse-grained simplified representation of the underlying data. In addition, the neural network representation and the phase-field equations filter out high-frequency noise in space and time without blurring interfaces (Fig. 2). These initial findings have triggered a new collaboration with the MPI for Dynamics of Complex Technical Systems, funded by the BiGmax network (see p. 82).

References

1. https://nomad-coe.eu/home-coe
2. Cautaerts, N.; Rauch, E. F.; Jeong, J.; Dehm, G.; Liebscher, C. H.: Scr. Mater. 201 (2021) 113930.
3. Cautaerts, N.; Crout, P.; Ånes, H.W.; Prestat, E.; Jeong, J.; Dehm, G.; Liebscher, C.H.; arXiv:2111.07347 (2021).
4. Wang, N.; Freysoldt, C.; Zhang, S.; Liebscher, C. H.; Neugebauer, J.: Microsc. Microanal. 27 (2021) 1454.

A microscopic view of electrochemical interfaces: *ab initio* molecular dynamics at controlled electrode potential

S. Wippermann[1], F. Deißenbeck[1], S. Surendralal[2], M. Todorova[2], J. Neugebauer[2]

[1]GO, [2]CM

Ever since the discovery of electricity, chemical reactions occurring at the interface between a solid electrode and an aqueous solution have aroused great scientific interest, not least by the opportunity to influence and control the reactions by applying a voltage across the interface. Our current textbook knowledge is mostly based on mesoscopic concepts, i.e. effective models with empirical parameters, or focuses on individual reactions decoupled from the environment, presenting therefore a serious obstacle for predicting what happens at a particular interface under particular conditions. In particular the current transition of the world to a more sustainable future requires a truly microscopic understanding of the electrochemical interface and double-layer by incorporating insights from the atomistic and electronic scale. Methodological breakthroughs at the MPIE during the last few years in both *ab initio* theory and experiments now allow us to directly characterise the structure and dynamics of electrified solid/liquid interfaces with atomic resolution at short timescales.

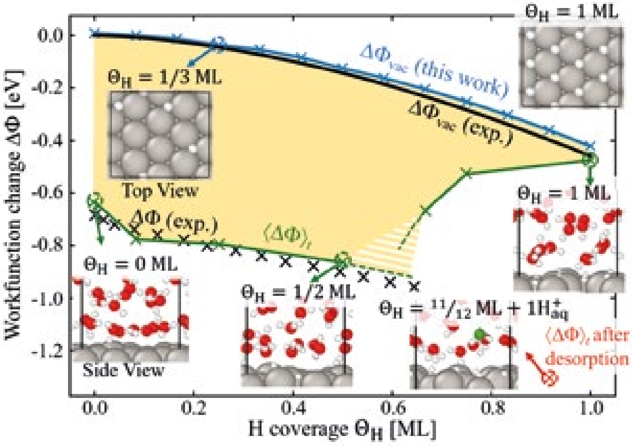

Fig. 1: *Work function (blue) and time-averaged electrode potential (green) as function of H coverage on Pt(111). For comparison, the experimentally measured work function (black line) and electrode potential (black crosses) are included. The critical H concentration is predicted to lie within the yellow hashed area, as as indicated by the extrapolated dashed green lines. Representative surface and interface structures at selected coverages are shown as insets.*

While the relation between the electrode potential and adsorbate coverage is accessible by experimental techniques such as cyclic voltammetry, understanding how exactly adsorbate interfacial structures influence the measured electrode potential evades direct experimental access. Consider the H-covered Pt(111)/H_2O interface, one of the best investigated "model" electrochemical systems due to its excellent catalytic performance for the hydrogen evolution reaction (HER). The onset of the HER coincides with the presence of a puzzling upper limit to the H adsorption at ≈ 0.66 ML (monolayer). A possible explanation is that water is not just a spectator but actively controls adsorption and chemical reactions at the metal-water interface.

To explore the impact of water on surface adsorption and the electrode potential, we perform a systematic comparison of the work function (electrode potential) of H-covered Pt(111) in vacuum (an aqueous environment) for coverages up to 1 monolayer employing density functional theory calculations [1]. Our calculations show an excellent agreement with experiment (Fig. 1) and allow us to directly explore how screening is realised at electrochemical interfaces. For the pristine Pt(111) interface we observe a large shift of the electrode potential with respect to the vacuum work function. This shift is caused by chemisorbed water molecules, which screen the interfacial dipole through charge transfer to the surface. Besides the Pt atom directly bonded to the H_2O molecule, this transfer involves several Pt atoms in its vicinity, effectively blocking neighbouring adsorption sites. Therefore, with the onset of H adsorption a competition arises between the water and hydrogen for the Pt atoms available for charge transfer. Increasing H coverage causes a monotonous loss of chemisorbed water, which is partially compensated by the charge transfer, due to H adsorption, but causes an increasing potential drop. Beyond a critical coverage of $\theta_H \approx 0.66$ ML this mechanism breaks down, because the increasing potential drop at the interface can no longer be screened by direct adsorption. Instead, the system responds by creating a large interfacial dipole through an electrochemical desorption reaction. This is achieved by deprotonation, where an H^+ ion forms in the vicinity of the interface, leaving its corresponding electron at the Pt(111) surface. The H^+ ion formation provides the necessary precursor state to enable the HER. Our findings directly link the existence of the breakdown in the electrode potential and the experimentally observed critical H adsorbate concentration at which hydrogen evolution starts.

In order to study electrochemical reactions -- such as, e.g., the HER -- in *ab initio* molecular dynamics simulations with explicit water, we build on our first principles electrochemical cell set-up introduced in the last reporting period [2], which allows us to actively control the local surface charge in order to drive the system towards the targeted value for the electrode potential. While the surface charge is constant in the thermodynamic limit, the microscopic size of the interfacial region targeted by our simulations requires treating the local surface charge as a thermodynamic degree of freedom.

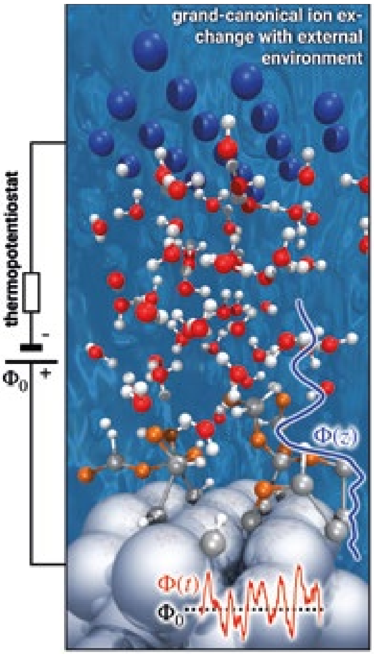

Fig. 2: *Electrochemical cell consisting of a Ge(100):H working electrode in contact with liquid water and a computational counter electrode (blue balls). Surface-adsorbed oxygen is marked in orange. In order to drive the system to the desired potential drop* Φ_0 *our thermopotentiostat algorithm controls the charge transferred between the two electrodes. Due to the microscopic size of the interfacial region targeted by our simulations, both the surface charge and the instantaneous potential* $\Phi(t)$ *are not constant but instead must be allowed to fluctuate. The time averaged potential* Φ_z *perpendicular to the interface is shown in blue.*

Changing the potential of a capacitive system, e.g. by controlling the surface charge in our electrochemical cell, is, however, a dissipative process. Energy is drained from molecular motions that are associated with a change in the electric dipole moment. This energy lost to potential control has to be compensated by corresponding thermal potential fluctuations, that exactly return the removed energy, on average, to those degrees of freedom where it was taken from. The physical system realises this by coupling to a fluctuating electric field created by temperature dependent charge fluctuations caused by the thermal motion of the electrons and ions.

To mimic this behaviour, our potentiostat must apply an electric field with an explicit finite temperature and hence become a thermopotentiostat. This can be achieved by adapting the fluctuation-dissipation theorem (FDT) to atoms exposed to an electric field. To this end, we used the FDT and the theory of stochastic differential equations in conjunction with Itō calculus. The derived direct expression for the electrode charge [2] is general, independent of the specific boundary conditions of the set-up and can be straightforwardly implemented in any molecular dynamics code, whether empirical or first principles. In fact, we have already implemented the thermopotentiostat in the VASP and lammps simulation packages (Fig. 2).

To demonstrate the performance of our thermopotentiostat approach we considered a topic that currently attracts a lot of attention. Recent experiments demonstrated that the presence of a solid-water interface appears to dramatically reduce the dielectric response of water from a highly polarizable medium down to a response that is close to the vacuum dielectric constant. We found that interfacial water forms stratified structures with a strongly modified dielectric behaviour, showing characteristic oscillations in the dielectric function close to interfaces and overscreening effects reminiscent of Friedel oscillations (see p. 125). As a consequence of the interfacial water's dielectric response, the potential drop between the electrode and the bulk water region occurs to a large degree directly at the interface, cf. blue line in Fig. 2. It is exactly within this region of interfacial water with modified properties where electrochemical reactions proceed.

Pioneered by Gerischer already 60 years ago, single-crystalline and atomically flat semiconductor surfaces interfaced with liquid water were popular model systems used to explore elementary electrochemical processes. The reaction mechanisms and their intermediate steps have been speculated about based on indirect evidence obtained from cyclic voltammetry and spectro-electrochemisty. We can now directly probe and predict the precise mechanistic details from first principles using our thermopotentiostat simulations. A corresponding snapshot for Ge(100) surfaces is shown in Fig. 2. Exceeding a potential drop of $\Phi_0 > 2$ V under anodic polarization, we observe that the reorientation of interfacial water is insufficient to screen the applied electric field. Instead, the interfacial dipole is screened by water dissociation, where H^+ is released into the solvent and OH^- groups adsorb on the surface (marked in orange). The subsequent discharge of the hydroxyl groups by the thermopotentiostat enables sustained reaction conditions for a continuous water splitting and electrooxidation of the surface.

These examples demonstrate how our continuous methodological advances help to reveal the elementary processes at electrified solid/liquid interfaces, enables us to show how idealised textbook models can be actually observed and revised, and thus achieve a truly microscopic understanding at the atomistic and electronic scales.

References

1. Surendralal, S.; Todorova, M.; Neugebauer, J.: Phys. Rev. Lett. 126 (2021) 166802.
2. Surendralal, S.: Todorova, M.; Neugebauer, J.: Phys. Rev. Lett. 120 (2018) 246801.
3. Deißenbeck, F.; Freysoldt, C.; Todorova, M.; Neugebauer, J.; Wippermann, S.: Phys. Rev. Lett. 126 (2021) 136803.

Part IV.

General information and statistics

Boards, directors, Max Planck fellows, and external scientific members

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Vice-president CPTS, Max-Planck-Society, Munich, Germany

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Steel Institute VDEh, Düsseldorf, Germany

Martin BAUES
Saarstahl AG, Völklingen, Germany

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ArcelorMittal Belgium N.V., Gent, Belgium

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Prof. Dr. Herman TERRYN
Vrije Universiteit Brussel (VUB), Brüssel, Belgium

Directors, Max Planck fellows, and external scientific members

Directors:

Prof. Dr. rer. nat. Gerhard DEHM, (since Oct. 2012)

Prof. Dr. rer. nat. Jörg NEUGEBAUER (since Nov. 2004)*

Prof. Dr.-Ing. Dierk RAABE (since July 1999)**

Prof. Dr. rer. nat. Martin STRATMANN (since Jan. 2000 / on leave for the time of his presidency of the MPG)

*Provisional Head of the Department Interface Chemistry and Surface Engineering

**Chief Executive since 29th Sept. 2010 (re-elected 2015 and 2020)

Max Planck fellows:

Prof. Dr. Jochen M. SCHNEIDER, Ph.D., RWTH Aachen University (Oct. 2015 - Sept. 2020)

Prof. Dr. Kristina TSCHULIK, Ruhr-Universität Bochum (since Sept. 2021)

External scientific members:

Prof. Dr. Mats HILLERT, Stockholm, Sweden

Prof. Dr. Reiner KIRCHHEIM, Göttingen

Guest scientists

Between January 2019 and November 2021 the Max-Planck-Institut für Eisenforschung GmbH hosted in total **158** international guest scientists.

Alexander von Humboldt Foundation

32 of our guests joined us with awards and fellowships from the Alexander von Humboldt Foundation.

- **25** Humboldt Research Fellowships for Postdoctoral Researchers
- **1** Capes-Humboldt Research Fellowship for Postdoctoral Researchers
- **4** Humboldt Research Fellowships for Experienced Researchers
- **1** Humboldt Research Award for outstanding academic record
- **1** Friedrich Wilhelm Bessel Research Award

In November 2021 the following Humboldt fellows were awarded who will join the MPIE in 2022:

- **4** Humboldt Research Fellowships for Postdoctoral Researchers
- **1** Humboldt Research Award

Also in November 2021, B. Gault and D. Raabe became **Humboldt scouts in frame of the Henriette Herz Scouting Programme**. Each scout has the opportunity to recommend three research talents from abroad for a Humboldt Research Fellowship. After formal approval, the fellowships will be granted directly.

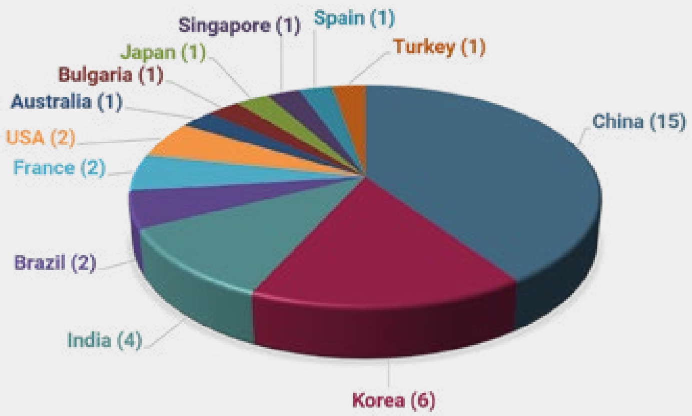
In the ranking of the Humboldt Foundation, the institute **was ranked first in Engineering Sciences** in 2020. It was also **ranked 3rd place over all sciences** of non-university research institutions.

Engineering Sciences

Absolute rankings	Institutions	Number of guest researchers per institution				Total number
		Fellows		Award winners		
		female	male	female	male	
1	MPI für Eisenforschung GmbH, Düsseldorf	5	25		3	33
2	MPI für Intelligente Systeme, Stuttgart		12	1	3	16
3	Forschungszentrum Jülich (FZJ)	4	7		1	12

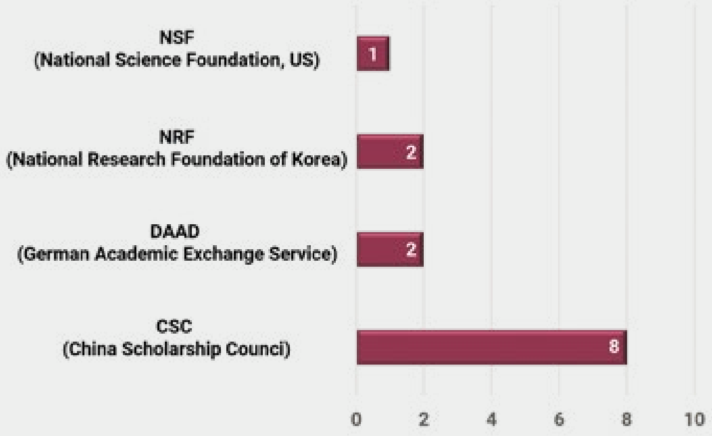
Source: https://www.humboldt-foundation.de/en/explore/figures-and-statistics/humboldt-rankings#c18797

Home countries of guests from the Alexander von Humboldt Foundation



Diverse fellowships

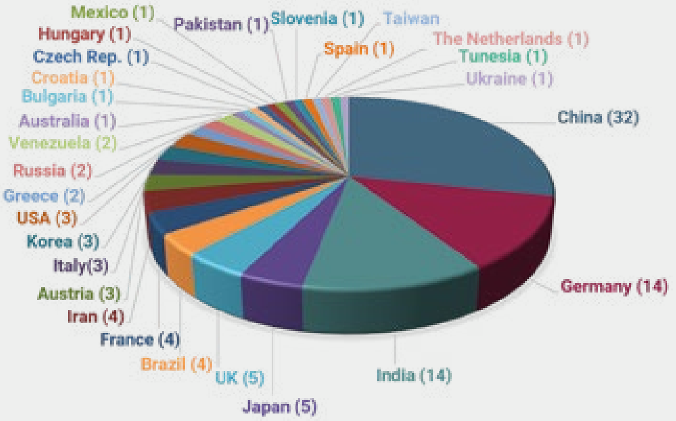
13 scientists came to the MPIE with diverse fellowships:



International research cooperation

With **113** scientists, the majority of our guest scientists stayed with us in frame of research cooperations. **27** of them being MPIE alumni.

Number of guest scientists per country



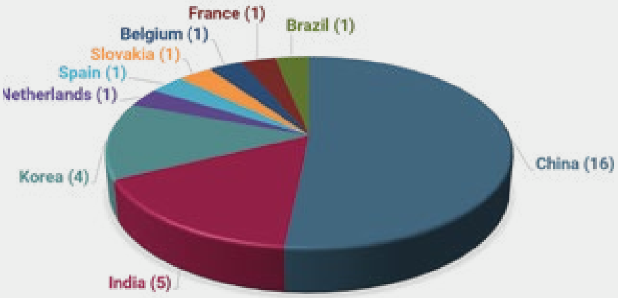
A detailed list of our "National and International Cooperation Partners" can be found from page 242 onwards.

MPIE guest programme

The MPIE offers outstanding international scientists possibilities to introduce their research topic at any level of their career within the status of a guest. At the same time, guest scientists are given the opportunity to exchange their ideas at an international level with excellent colleagues, thereby establishing important scientific networks.

During 2019-2021 a total of **31** postdoctoral researchers joined the MPIE in frame of the guest programme with a duration of 12-24 months.

Home countries of postdocs in frame of the MPIE guest programme



Scientific honours & appointments

Honours

2018 (not included in the Report 2016 – 2018)

A. Dutta: Winner German Science Slam Championship

F. Roters: Elected to the executive board of the German Materials Society (DGM)

2019

L. Abdellaoui: Graduate Student Conference Registration Wavier for the 38th International Conference on Thermoelectrics

R. Changizi: 1st prize "Arts in Science" image competition, "Microscopy Conference 2019"

A. Dutta: Winner European Science Slam Championship

T. Gänsler: 2nd prize "Electron Microscopy – Physical Sciences" category, image competition of the Royal Microscopical Society

A. Kwiatkowski da Silva: Selected participant in the 69th Nobel Laureate Meeting

N. Peter: Selected participant in the 69th Nobel Laureate Meeting

D. Raabe: Chair of the Gordon Research Conference on Physical Metallurgy at the University of South New Hampshire in Manchester (USA)

D. Raabe: KAIST Lecture Series in Materials Science and Engineering 2019 at the Korea Advanced Institute of Science and Technology KAIST (Korea)

D. Raabe: Bauerman Lecture Award 2019, Imperial College London, Royal School of Mines

I. Souza Filho: Brazilian PhD Award 2019

H. Springer: Friedrich Wilhelm Preis, RWTH Aachen University

M. Stratmann: Re-elected President of the MPG

M. Stratmann: Honorary Member of the Indian Institute of Metals (IIM)

2020

B. Gault: Gottfried Wilhelm Leibniz Award 2020, German Research Foundation (DFG)

MPIE: 1st place in the Alexander von Humboldt Ranking in Engineering Sciences among all non-university research institutions & 3rd place overall sciences of non-university research institutions

C. Scheu: Elected to the Review Board of the German Research Foundation (DFG)

F. Roters: Re-elected to the executive board of the DGM

H. Zhao: ICAA (International Conference on Aluminium Alloys) Early Career Researcher Award

H. Zhao: TMS Light Metals Subject Award - Aluminum Alloys

S. Zwaka: International Research Marketing Prize, German Research Foundation (DFG)

2021

S. Antonov: Postdoctoral Student Award & the Young Leaders International Scholar - JIM, The Japan Institute of Metals and Materials (JIM) and the Federation of European Materials Societies (FEMS)

R. Aymerich Armengol: Selected participant for the 70th Interdisciplinary Lindau Nobel Laureate Meeting

R. Aymerich Armengol, R. Changizi, A.A. El-Zoka, R. Hosseinabadin and E.I. Saad: Winners of MPIE Science Video Competion Prize

S.H. Kim: Müller Emerging Scientist Award at the Atom Probe Tomography and Microscopy (APT&M) conference

Y. Ma: Walter Benjamin Grant, German Research Foundation (DFG)

Career programmes for female scientists

B. Bellon: Selected to participate in the MPG Sign Up! For Your Career: Earlier postdoc phase in 2022

A. Garzon: Selected to participate in the MPG Sign Up! For Leadership in 2022

O. Kasian: Selected to participate in the MPG Sign Up! Career building for female post docs in 2019

C. Liu: Selected to participate in the MPG Sign Up! For Leadership in 2022

A. Mahajan: Selected to participate in the MPG Sign Up! For Your Career: Earlier postdoc phase in 2022

M. Todorova: Accepted for the Boost! Programme of the MPG in 2020

Fellowships & scholarships

2019

L. Gomell: PhD scholarship of the German Academic Scholarship Foundation

P.-Y. Tung: Taiwanese Government Scholarship to Study Abroad (GSSA)

2020

Z.Wang: FP-RESOMUS Fellowship (Fellowship Program of the NCCR MUST and the Cluster of Excellence RESOLV)

Several of our guest scientists - including all fellows from the Alexander vonHumboldt Foundation - also joined the MPIE with fellowships and schlorships. Please see page 230 onwards for more information.

MPIE trainees

2018 (not included in the Report 2016 – 2018)

T. Freieck: "Chambers best 2018" in the field of materials testing, IHK (chamber of industry and commerce) Düsseldorf (Germany)

2020

A. Laimmer: Max Planck Trainee Award 2020 of the Max Planck Society (Germany)

A. Laimmer: "Chambers best 2020" in the field of materials testing by the IHK (chamber of industry and commerce) Düsseldorf (Germany)

2021

H. Bögershausen, H. Faul, A. Schulz: First place at the Innovationssemester competition of the Wissensregion Düsseldorf e.V.

S. Sprengel: Max Planck Trainee Award 2021 of the Max Planck Society (Germany)

Paper, poster, excellence in reviewing, travel and communication awards

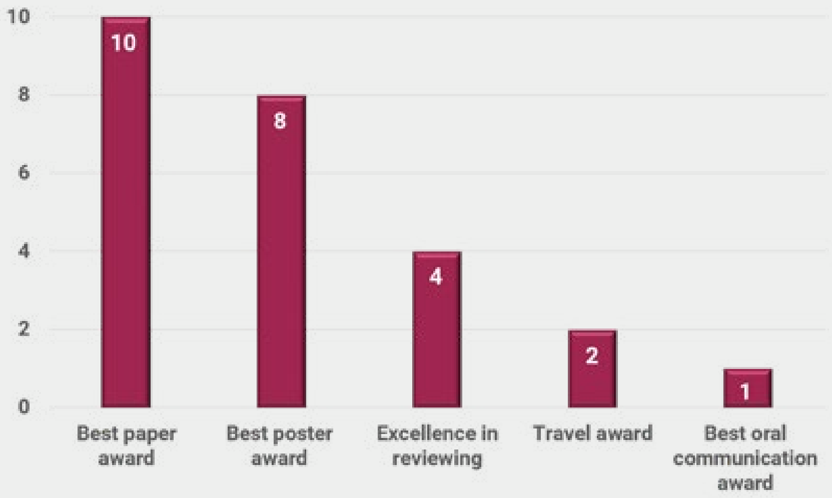
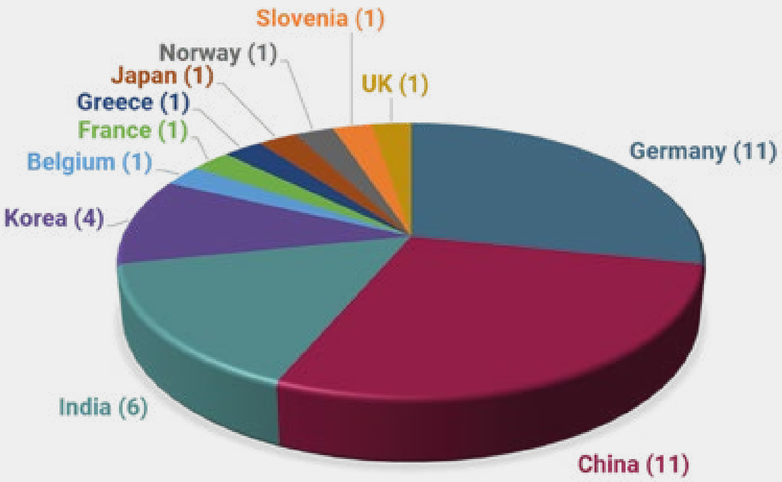


Fig.1: *Summary of all best paper, best poster, excellence in reviewing, travel and communication awards received by MPIE scientists between 2019 – 2021. These awards are not listed above.*

Appointments

Destination countries of appointments



2018 (not included in Scientific Report 2016 - 2018)

B. Gault: Assistant Professor at the Imperial College London, UK

J. Han: Assistant Professor, Department of Materials Science and Engineering, Chungnam National University, South Korea

O. Kasian: Helmholtz Young Investigator Group at the Helmholtz-Zentrum Berlin für Materialien und Energie, Germany

J. Zavašnik: Member of the Gaseous Electronics Department in the Jožef Stefan Institute (JSI) and Assistant Professor at the Jožef Stefan International Postgraduate School in Ljubljana, Slovenia

2019

H.-O. Fabritius: Full Professor of Bionik und Materialentwicklung, University of Applied Science Hamm-Lippstadt, Germany

X. Fang: Head of the Dislocation Based Functionalities Group within the Ceramics Group in the Department of Materials and Earth Sciences at the Technical University of Darmstadt

P.K. Gokuldoss: Assistant Professor, Indian Institute of Technology Madras, India

J.G. Kim: Assistant Professor, Department of Materials Engineering and Convergence Technology, ReCAPT, Gyeongsang National University, Korea

M. Koyamna: Assistant Professor, Tohoku University, Japan

A. Lahiri: Assistant Professor, Metallurgical and Materials Engineering Department, Indian Institute of Technology Roorkee, India

Z. Li: Full Professor, School of Materials Science and Engineering, Central South University, China

W. Lu: Professor, Central South University, China

S.K. Makineni: Assistant Professor, Department of Materials Engineering, Indian Institute of Science, Bangalore, India

Ashish Kumar Saxena: Assistant Professor in the Centre for Innovative Manufacturing Research (CIMR) in the Vellore Campus of the Vellore Institute of Technology (VIT), India

S.S. Sohn: Assistant Professor, Korea University

H. Springer: Professorship at the RWTH Aachen University, Academic and Research Department Metallic Composites, Germany

A. Tripathi: Assistant Professor, Department of Metallurgical and Materials Engineering, Malaviya National Institute of Technology Jaipur, India

Y.Yao: Full professor at the School of Mechanics, Civil Engineering and Architecture at the Northwestern Polytechnical University (NWPU) in Xi'an, Shaanxi, China

2020

S. Brinckmann: Permanent head of the Micromechanics and Tribology Group at the Forschungszentrum Jülich GmbH, Germany

M. Diehl: Full Professor, Katholieke Universiteit Leuven, Department of Computer Science, Department of Materials Engineering, Belgium

H. Fan: Professor, Associate Chair, Department of Mechanics, Sichuan University, China

M. Ghidelli: Permanent staff scientist position in the Laboratoire des Sciences des Procédés et des Matériaux (LSPM) at the Université Sorbonne Paris Nord (CNRS), France

W. Guo: Associate Research Fellow in the Research Institute of Aero-Engine at Beihang University in Beijing, China

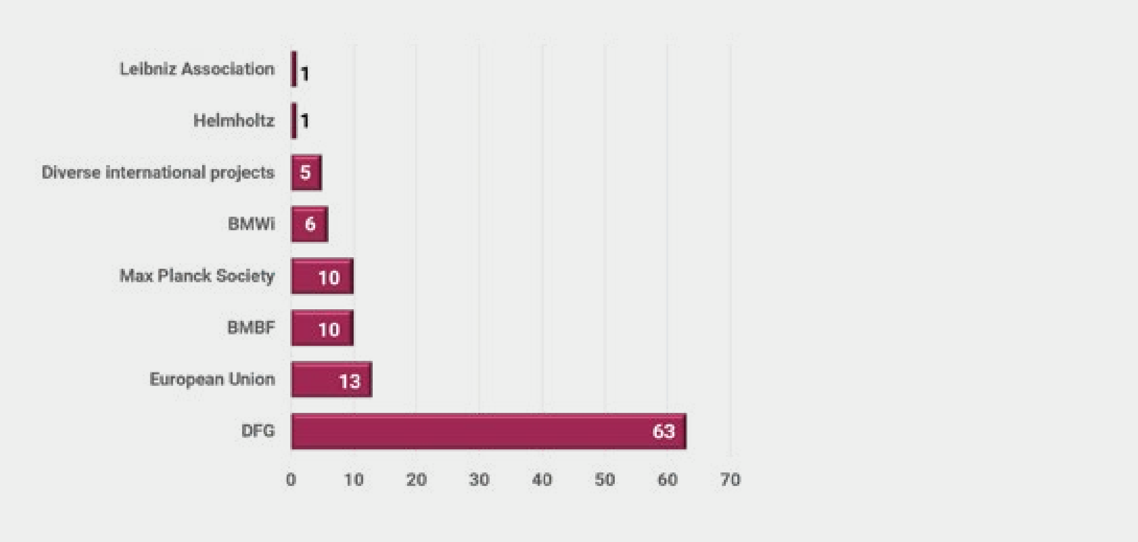
E. Jägle: Univ.-Prof., Fakultät für Luft- und Raumfahrttechnik Universität der Bundeswehr München, Germany

C. Kirchlechner: Head of the Institute of Biomechanics and Materials at the Karlsruhe Institute of Technology (KIT), Germany

M. Ledendecker: Independent Group Leader at the TU Darmstadt, Germany

D.-H. Lee: Professor, Chungnam National University, Korea

Participation in research programmes 2019 – 2021



National

BMBF

DaNiSh - Development of a high gamma nickel base superalloy with outstanding material properties for applications in drive technology and turbomachinery

MANGAN - Mechanistic investigations of electrochemical oxygen evolution on model electrodes

MaterialDigital - Joint project: Innovation platform Material Digital - sub-project: Software tools

NanoSolar - Semiconducting nanocomposites with tailored optical and electronic properties

Optimization of the efficiency of solar cells based on 3-dimensional chemical analyses on an atomic scale

PrometH2eus - Project network for optimised material development for technical H_2 generation through improved oxygen electrodes

RAVE-K - Resource-saving assembly and connection technology for low-voltage technology contact materials containing precious metals

Stahl*Digital* - Workflows und Plattformintegration

UGSLIT - Ultra high-strength weight-reduced steels for resource-saving lightweight construction in transport applications

White-Etching-Cracks - Knowledge-based design of wear resistant bearing steels by atomic-scale characterization as countermeasure against white-etching crack failure in wind power plants

BMWi

H2BS - New hydrogen barrier coatings for low-cost and high-strength steels in hydrogen technology

MAXCoat - Development of a cost-efficient ferritic bipolar plate with MAX phase corrosion protection for applications in portable and mobile fuel cells

MODUL - Integrated material and process development of high-modulus steels

PAULL - Activation of ultra-long lifetimes of fuel cells; Subproject: Elucidation of cross-scale degradation mechanisms during electron microscopic characterization of MEAs before and after operation

Pro-FeAl - Process development for economical, efficient turbine components made of iron aluminides (Fe-Al), Subproject C: Alloy design and characterisation

PtTM@HGS - Development of cost-efficient, high performance gas diffusion electrodes for polymer electrolyte membrane fuel cells (PEM-FC) with low Platinum loading and novel hollow graphitic spheres as support

DFG

DFG awards

Gottfried Wilhelm Leibniz Award 2020 for B. Gault

DFG collaborative research centres (SFB), transregio projects (TRR) & priority programmes (SPP)

SFB 761: Steel - Ab Initio. Quantum Mechanics Guided Design of New Fe-based Materials

- A 02: Ab initio thermodynamics und kinetics in the Fe-Mn-Al-C system
- A 07: Microstructure mechanics and fundamentals of concurrent twinning and martensite formation
- A 09: Ab initio based mesoscale simulation of hydrogen embrittlement
- C 01: Microstructure analytics
- C 04: Fatigue, damage and stress corrosion cracking under cyclic loading
- C 05: Defects and Stresses in Fe-Mn-C Steels
- C 08: 3D atomic analysis of the local chemical composition by atom probe tomography
- C 10: Deformation behavior of multi-phase steels
- T 07: Liquid metal embrittlement by Zn in Mn-containing steels with significant austenite content

SFB 1232: From colored states to evolutionary structural materials

- S02: Correlative study towards experimental validation of the high throughput methodology

SFB 1394: Structural and Chemical Atomic Complexity: From Defect Phase Diagrams to Material Properties

- A01: Solid solution effects on dislocation activity
- A02: Atomistic simulations of dislocation processes
- A06: Ab initio accuracy on large scales using machine learning
- B01: Interfaces in Mg-Al-Ca alloys in dependence on chemical composition and temperature
- B04: Corrosion of Mg alloys and intermetallics
- B06: Fracture of complex intermetallic phases: influence of temperature
- C01: Multiphysics description of Mg composites at the grain scale
- C05: Ab initio thermodynamics of defect phases

SFB/ TRR 188: Damage Controlled Forming Processes

- B03: Position resolved damage nucleation and growth at the microstructure length scale

TRR 103: From Atoms to Trubine Blades – A Scientific Approach for Developing the Next Generation of Single Crystal Superalloys

- A04: Investigation of the local alloy composition by atom probe tomography

TRR 270: HoMMage - Hysteresis design of magnetic materials for efficient energy conversion: HoMMage

Priority programmes

SPP 1239: Modification of Microstructure and Shape of Solid Materials by External Magnetic Fields: Ab initio investigation of temperature dependent effects in magnetic shape memory Heusler alloys

SPP 1568: Design and Generic Principles of Self-healing Materials

- Towards self-healing metals by employing optimally-dispersed Ti-Ni Shape-memory nano-particles

SPP 1594: Topological Engineering of Ultra-Strong Glasses

- Quantum mechanically guided design of ultra-strong and damage-tolerant glasses

SPP 1613: Regeneratively Produced Fuels by Light Driven Water Splitting: Investigation of Involved Elementary Processes and Perspectives of Technologic Implementation

- Metal oxide nanostructures for electrochemical and photoelectrochemical water splitting

SPP 1713: Strong Coupling of Thermo-Chemical and Thermo-Mechanical States in Applied Materials

- Evolution of strengthening phases under in-service stresses and temperatures: phase-field and experimental study
- Mechano-chemical coupling during precipitate formation in Al-based alloys
- Phase-field-based chemomechanical models for phase transitions and dislocation-microstructure interaction in metallic alloys with application to kappa-carbides
- Thermo-chemo-mechanical coupling during thermomechanical processing of microalloyed steels

SPP 1839: Tailored Disorder - A science- and engineering-based approach to materials design for advanced photonic applications: Broadband reflecting fibers with tailored structures inspired by desert ants

SPP 1959: Manipulation of matter controlled by electric and magnetic fields: Towards novel synthesis and processing routes of inorganic materials

- Electro-plasticity in Al-Cu eutectic alloys
- Micromechanisms of the electro-plastic effect in magnesium alloys investigated by means of electron microscopy

SPP 2006: Compositionally Complex Alloys – High Entropy Alloys (CCA – HEA)

- MarioCCArt - Mechanical properties and hydrogen tolerance of particle-reinforced CCA produced by additive manufacturing
- MULTI TRIP CCA-s – Design and mechanical properties of compositionally complex alloys from twinning-induced towards bidirectional transformation-induced plasticity
- PaCCman - Particle-strengthened Compositionally Complex Alloys - interlinking powder synthesis, additive manufacturing, microstructure evolution and deformation mechanisms
- Tailored precipitation (B2, $L2_1$) strengthened, compositionally complex FeAlCr (Mn, Co, Ni, Ti) alloys for high temperature applications

FOR 1700: Metallic Nanowires on the Atomic Scale: Electronic and Vibrational Coupling in Real World Systems

DFG research grants

Ab initio based calculation of the stability of selected TCP precipitates in steels: Temperature and interface effects

Ab initio determination of free energies and derived properties (heat capacities, vacancies, solvus boundaries) for selected Al alloys containing Si, Mg and Cu

Additive Manufacturing - Precipitation kinetics during non-linear heat treatment in Laser Additive Manufacturing

AHEAD - Analysis of the Stability of High Entropy Alloys by Dewetting of Thin Films

Automated analysis and validation of interatomic potentials for application in Materials Science

Can high strength and moderate ductility be combined in wear resistant coatings? A fundamental plasticity study of X_2BC nanolaminates (X=Hf, MO)

Characterization and modeling of the interplay between grain boundaries and heterogeneous plasticity in titanium

Consistent physically-based modeling of dynamic recrystallization under hot working conditions

CORRKEST - Correlative Characterization of co-evaporated $CU_2ZnSnSe_4$- thin films

Correlation of growth, structure, optical and electronic properties of novel $Nb_3O_7(OH)$ and NB_2O_5 nanostructures

C-Tram - Atomic scale redistribution of carbon during the transformation from austenite to martensite in steels

Diffusion in high entropy alloys: Development and application of an experiment-ab initio approach

Elementary mechanisms of tribologically-induced oxidation in copper

Exploring Multinary Nanoparticles by Combinatorial Sputtering into Ionic Liquids and Advanced Transmission

Electron Microscopy

Fracture initiation in FCC and BCC metals during tribology

From interatomic potentials to phase diagrams: Integrated tools for validation and fitting

Hydrogen-microstructure interactions in iron-based alloys at small scales: from amorphous, via nanocrystals, to polycrystals

MAGIKID - Magnetism in iron alloys: thermodynamics, kinetics and defects

Metallic glasses - Structure, phase formation and properties of metallic glasses manipulated by electric current

Mxene – Mxene-based energy materials guided by 3D Atomic-Resolution Tomography

RE-MAP - Rare-earth based alloys for hard-magnetic applications: Temperature and pressure dependent phase stabilities

Spektralmethoden / KugelRVE - Spectral methods for spherical representative volume elements

Structure, phase formation and properties of metallic glasses manipulated by electric current

Study of grain-boundary-dislocation interactions by advanced in situ µLaue diffraction

XMicroFatigue - X-ray Laue Microscopy to Understand Fatigue Damage

DFG cluster of excellence

Stability of electrode materials in an electrochemical environment", DFG Cluster of Excellence 1069 RESOLV (Ruhr Explores Solvation)

DFG competition on international research marketing

Coffee with Max Planck: Research Opportunities at the MPIE

Max Planck Society

Combinatorial design of novel rare-earth free, high-entropy based permanent magnets

Designing Damage Tolerant Functional Oxide Nanostructures: Damage Tolerance Studies on Barium Titanate at Small Length Scales

Environmental Degradation of High-Temperature Materials in Service Conditions

International Max Planck Research School for Interface Controlled Materials for Energy Conversion IMPRS SurMat (IMPRS - SurMat)

MaxNet BiGmax - Max Planck Research Network on big-data-driven material science

BiGmax project: Investigation of deep learning-based approaches to solve partial differential equations in mechanical equilibrium problems

Max Planck Fellow Group Electrochemistry & Nanoscale Materials (Kristina Tschulik)

Max Planck Fellow Group Self-Reporting Materials (Jochen Schneider)

Microstructure Design of High-Performance Materials for Harsh Energy Applications

Stress and defects driven phase transformations

Helmholtz

Helmholtz school for data science in life, earth, and energy (HDS-LEE)

Machine Learning Enhanced Multi-Scale Modeling of Ice Deformation and Melting

Leibniz Association

CarMON - New Carbon-Metal Oxide Nanohybrids for Efficient Energy Storage and Water Desalination

International

European Union

ERC (European Research Council)

GB-CORRELATE - Correlating the State and Properties of Grain Boundaries

SHINE - Seeing hydrogen in matter

TIME-BRIDGE - Time-scale bridging potentials for realistic molecular dynamics simulations-TIME-BRIDGE

Marie Skłodowska-Curie Actions

smartCC - Designing novel smart sensor interfaces based on a biologically abundant peptide motif

FP-RESOMUS - Fellowship Program of the NCCR MUST (National Competence Center for Research in Molecural Ultrafast Science and Technology) and the Cluster of Excellence RESOLV-FP-RESOMUS

Collaborative projects

ADVANCE - Sophisticated experiments and optimisation to advance an existing CALPHAD database for next generation TiAl alloys

FORGE - Development of novel and cost-effective coatings for high-energy processing applications

H2Free - Investigation and modelling of hydrogen effusion in electrochemically plated ultra-high-strength-steels used for landing gear structures

UltimateGaN - Technologies for novel power electronics and radar applications; Subproject: Identification of degradation and failure mechanisms and optimisation of novel nitide-based power electronics devices by ab initio simulations

RFCS (Research Fund for Coal and Steel)

DELIGHTED - Design of Lightweight Steels for Industrial Applications

HYDRO-REAL - Hydrogen Interaction with Retained Austenite Under Static and Cyclic Loading Conditions

MicroCorr - Improving steel product durability through alloy coating microstructure

MuSTMeF - Multi Scale Simulation Techniques for Metal Forming

FFG (Austrian Research Promotion Agency)

Fundamentals and tools for integrated computational modeling and experimental characterization of materials in the atomic to micrometer scale range”, Project A1.23 (COMET II) / Project P1.1 (COMET III)

FWO - Fonds Wetenschapelijk Onderzoek

PredictCor - Knowledge and technology platform for prediction of durability and lifetime of organic coated metals under long-term environmental corrosion

M2i

DENS - Digitally Enhanced New Steel Product Development

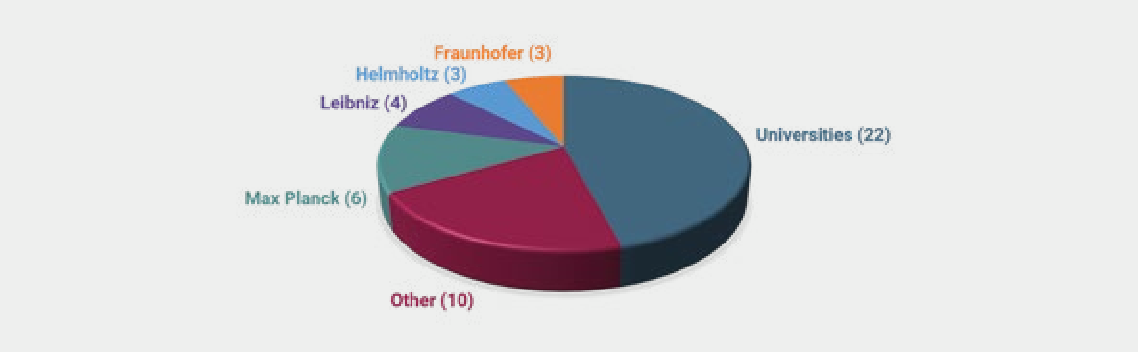
- T17019o “Full field simulation of Dynamic Recrystallization”
- T17019e “Multi-field RVE simulation”

NWO (Dutch Research Council)

Systematic simulation of high-entropy alloys

Collaboration with national and international universities and research institutes

National



Universities

Brandenburgische Technische Universität Cottbus-Senftenberg

Bergische Universität Wuppertal

Friedrich-Alexander-Universität Erlangen-Nürnberg

Heinrich-Heine-Universität Düsseldorf

Karlsruher Institut für Technologie - KIT

Ludwig-Maximilians-Universität

Otto-von-Guericke-Universität Magdeburg

Ruhr-Universität Bochum

RWTH Aachen

Technische Universität Bergakademie Freiberg

Technische Universität Clausthal

Technische Universität Darmstadt

Technische Universität München

Universität Bayreuth

Universität Bielefeld

Universität Bremen

Universität Duisburg-Essen

Universität Konstanz

Universität Paderborn

Universität Siegen

Universität Stuttgart

Westfälische Wilhelms-Universität Münster

Research institutes

Access e. V.

Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin

Deutsche Akademie der Naturforscher Leopoldina, Halle

Deutsches Zentrum für Luft- und Raumfahrt (DLR), Bonn

fem Forschungsinstitut Edelmetalle + Metallchemie, Schwäbisch Gmünd

Forschungszentrum Jülich GmbH

Institut für Angewandte Informatik e.V. (InfAI), Leipzig

Institut für Energie- und Umwelttechnik e.V. (IUTA), Duisburg

Fraunhofer Institut für Lasertechnik (ILT), Aachen

Fraunhofer Institut für Werkstoffmechanik (IWM), Freiburg

Fraunhofer-Institut für Werkstoff- und Strahltechnik (IWS), Dresden

Fritz-Haber-Institut der Max-Planck-Gesellschaft, Berlin

Helmholtz-Institut Erlangen-Nürnberg (HI ERN) für Erneuerbare Energien, Erlangen

Helmholtz-Zentrum Berlin (HZB) für Materialien und Energie, Berlin

Helmholtz-Zentrum Hereon: Institut für Werkstoffphysik, Geesthacht

Leibniz-Institut für Festkörper- und Werkstoffforschung (IFW), Dresden

Leibniz-Institut für Neue Materialien (INM), Saarbrücken

Leibniz-Institut für Plasmaforschung und Technologie e.V. (INP), Greifswald

Leibniz Institut für Werkstofforientierte Technologien (IWT), Bremen

Max-Planck-Institut für Chemische Physik fester Stoffe, Dresden

Max-Planck-Institut für Festkörperforschung, Stuttgart

Max-Planck-Institut für Intelligente Systeme, Stuttgart

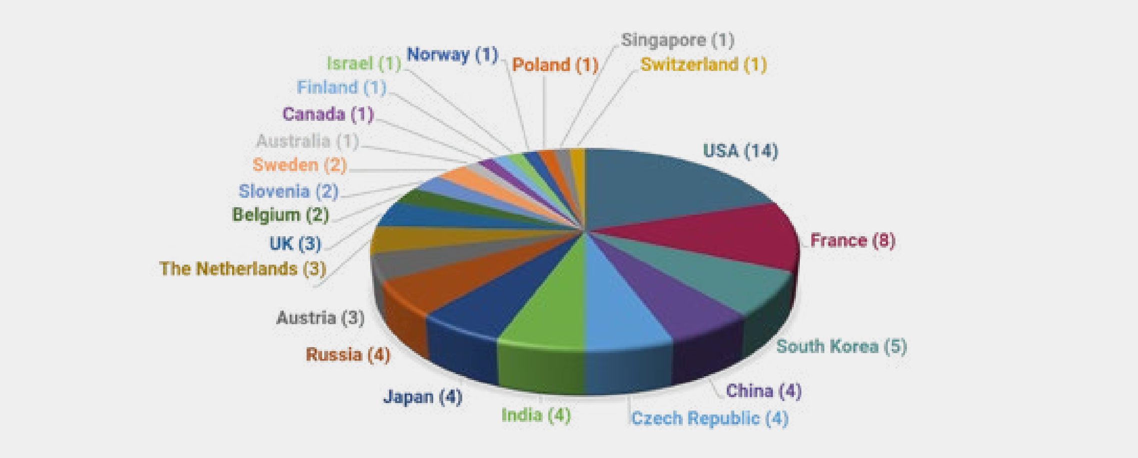
Max-Planck-Institut für Kohlenforschung

Max-Planck-Institut für Chemische Energiekonversion

VDEh-Betriebsforschungsinstitut GmbH (BFI), Düsseldorf

Zentrum für Brennstoffzellen-Technik GmbH, Duisburg

International



Universities

Aix-Marseille University, France

Beijing Institute of Technology, China

Belgorod State University, Russia

Brown University, Providence, Rhode Island, USA

Central South University, Changsha, China

Charles University Prague, Czech Republic

Clemson University, South Carolina, USA

Delft University of Technology, The Netherlands

Ecole Polytechnique, France

Hasselt University, Belgium

Hokkaido University of Science, Sapporo, Japan

Imperial College London, UK

Indian Institute of Technology Bombay, India

Indian Institute of Technology Chennai, India

Indian Institute of Technology Madras, India

Kangwon National University, Chuncheon, South Korea

Katholieke Universiteit Leuven, Belgium

Korea University, Seoul, South Korea

KTH Royal Institute of Technology, Stockholm, Sweden

Leiden University, The Netherlands

Linköping University, Sweden

MINES ParisTech, PSL - Research University, France

Montanuniversität Leoben, Austria

National University of Science and Technology (MISIS), Moscow, Russia

North Carolina State University, Raleigh, North Carolina, USA

Northwestern University, Illinois, USA

Norwegian University of Science and Technology (NTNU), Trondheim, Norway

Silesian University of Technology, Gliwice, Poland

Singapore University of Technology and Design, Singapore

Southern University of Science and Technology, Shenzhen, China

Tampere University, Finland

Technical University Eindhoven, The Netherlands

Technical University Liberec, Czech Republic

Tongji University, Shanghai, China

Université Sorbonne Paris Nord, France

University of British Columbia, Vancouver, Canada

University of California, Berkeley, California, USA

University of California. Santa Barbara, California, USA

University of California, Los Angeles, California, USA

University of Florida, Gainesville, USA

University of Illinois, USA

University of Manchester, UK

University of Oxford, UK

University of Ulsan, South Korea

University of Vienna, Austria

University of West Bohemia, Pilsen, Czech Republic

UNSW Sydney, Australia

Research institutes

Academy of Sciences of the Czech Republic, Brno, Czech Republic

Austrian Academy of Sciences, Leoben, Austria

CEA Saclay, Paris, France

Centre de Mise en Forme des Matériaux (CEMEF), Nice, France

Centre National de la Recherche Scientifique (CNRS): Office National d'Etudes et de Recherches Aérospatiales (ONERA), Châtillon, France

Council of Scientific and Industrial Research, National Metallurgical Laboratory (CSIR-NML), Burmamines, Jamshedpur, India

Empa – The Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland

Institut de Physique et Chimie de Matériaux de Strasbourg, France

Institute for Metals and Technology (IMT), Ljubljana, Slovenia

Institute of Quantum Materials Science, Yekaterinburg, Russia

Jožef Stefan Institute, Ljubljana, Slovenia

Korea Advanced Institute of Science and Technology (KAIST), Daejeon, South Korea

Korea Institute for Industrial Technology, Busan, South Korea

Lawrence Berkeley National Laboratory: Molecular Foundry, National Center for Electron Microscopy, Berkeley, USA

Lawrence Livermore National Laboratory, Livermore, CA, USA

Los Alamos National Laboratory, New Mexico, USA

Massachusetts Institute of Technology (MIT), Cambridge, Massachusetts, USA

National Institute for Materials Science (NIMS), Tsukuba, Japan

National Institute of Advanced Industrial Science and Technology (AIST), Tokyo, Japan

Oak Ridge National Laboratory, Oak Ridge, Tennessee, USA

Skolkovo Institute of Science and Technology, Moscow, Russia

Technion – Israel Institute of Technology, Haifa, Israel

Tokyo Institute of Technology, Japan

Collaborating industrial partners and patents

National

BMW Group AG

Deloro Wear Solutions GmbH

DHS – Dillinger Hütte Saarstahl AG

Dr. Kochanek Entwicklungsgesellschaft

Freudenberg Sealing Technologies, Freudenberg FST GmbH

Heraeus Deutschland GmbH & Co. KG

Infineon Technologies AG

Innovationsgesellschaft für fortgeschrittene Produktionssysteme GmbH (inpro)

KSB Stiftung

Leistritz Turbinentechnik GmbH

Materials Science International Services GmbH (MSI)

MTU Aero Engines AG

Otto Junker GmbH

point electronic GmbH

Robert Bosch GmbH

Rolls-Royce Deutschland Ltd & Co KG

Salzgitter Mannesmann Forschung GmbH

Schott AG

Siemens AG

Speira GmbH, Research & Development

thyssenkrupp Steel Europe AG

VDM Metals International GmbH, Research and Development

Volkswagen AG

International

AMAG Austria Metall GmbH, Austria

Alemnis, Switzerland

Denso Corporation KK, Japan

ELHCO - Electroless Hard Coat S.A., Spain

Exaddon AG, Switzerland

Infineon Technologies Austria AG, Austria

JFE Steel Corporation, Japan

KAI Kompetenzzentrum Automobil- und Industrieelektronik GmbH, Austria

Kobe Steel, Ltd., Japan

Materials Center Leoben Forschungs GmbH, Austria

Rolls-Royce Motor Cars Ltd, England

Sandvik Coromant R&D, Sweden

Tata Steel Nederland Technology BV, The Netherlands

Thermo-Calc Software AB, Sweden

Vallourec Research Center France, France

Wärtsila Finland Oy, Finland

Winterthur Gas & Diesel Ltd, Switzerland

Patents

Patents issued in the given time schedule

Date of Issue	Description	Inventors
utility patent from August 18, 2016, prolongated August 31, 2019 until August 2022	**Multi purpose method cell** (20 2016 104 543.6)	Kerger, Philip Rohwerder, Michael
January 28, 2020 June 06, 2021	**Process for recycling of noble metals** USA 15/771,500 EP 3368698	Mayrhofer, Karl Hodnik, Nejc Baldizzone, Claudio

Patents filed in the given time schedule

Date of Pending	Description	Inventors
October 9, 2018	**Temperature Measurement for a High Temperature Micro-deformation Device** (EP 18199374.2.)	Arigela, Viswanath Gowtham Kirchlechner, Christoph Gonzalez, Iwan Kölling, Michael

Patents - overview

Date of issue	Description	Inventors
June 05, 2015	**Korrosionsbeständiger TWIP-Stahl** (JP 2011-553279.)	Weber, Sebastian Mujica Roncery, Lais
June 07, 2018	**Fabrication of nanoporous carbide membranes** Joint invention with Universitat Politecnica de Catalunya (EP 2 664 683)	Renner, Frank Duarte-Correa, Maria Jazmin Lengsfeld, Julia Bruna, Pere, Barcelona Tech
March 03, 2016	**Nanoelektroden-Partikelfalle für empfind-liche spektroskopische und elektronische Analyse** Joint invention with Academia Sinica Taipe, who is apllicant (TWI490487)	Erbe, Andreas Chia-Fu Chu Ming-Li Chu Leonardo Lesser-Rojas, all Academia Sinica, Taipe
January 8, 2015	**Schichtsystem zum Korrosiosschutz ASKORR** Joint invention with MPI für Polymerforschung and Fraunhofer Gesellschaft (DE 10 2012 209 761.9)	Rohwerder, Michael Vimalanandan, Ashokanand Tran, The Hai Landfester, K. Crespy, D. Fickert, J., MPI für Polymerfoschung
October 25, 2016 October 27, 2015	**Process for recycling of noble metals** (DE 102015118279.3) (PCT/ DE2016/ 100498; EP 3 368 698)	Mayrhofer, Karl Hodnik, Nejc Baldizzone, Claudio
January 31, 2013 May 19, 2009	**Hochfester C-Stahl mit Superplastizität** Joint invention with Daimler Chrysler (DE 10 2005 027 258.4) (US 7.534314B2)	Frommeyer, Georg Gerick, Arndt Haug, Tilmann, Kleinekathöfer, Wolfgang, all DaimlerChrysler AG

Conferences, symposia and meetings organized by the institute

2018 (not included in the Scientific Report 2016 - 2018)

L. Stephenson co-organized and chaired a session at the "International Microscopy Conference (IMC)", Sydney (Australia), 09 – 14 Sep 2018

L. Stephenson co-organized the "European Atom Probe Workshop (EAPW)" at the MPIE, Düsseldorf (Germany), 07 – 09 Nov 2018

2019

F. Stein organized the "International Workshop on Laves Phases" at the MPIE, Düsseldorf (Germany), 14 - 15 Jan 2019

S. Brinckmann, *G. Dehm*, and *C. Scheu* organized the Symposium "Mechanics Meets Energy 2019" in Cuxhafen (Germany), 21 - 25 Jan 2019

F. Stein and *M. Palm* co-organized the "3rd MSIT Winter School on Materials Chemistry" at Ringberg Castle, Kreuth (Germany), 03 - 06 Mar 2019

F. Körmann co-organized the Topical Session "High entropy and compositionally complex alloys" at the DPG Spring Meeting 2019 in Regensburg (Germany), 01 - 05 Apr 2019

C. Liebscher, *F. Körmann*, *B. Grabowski*, and *S. Divinski* organized the symposium "High entropy and compositionally complex alloys" at the DPG Spring Meeting 2019 in Regensburg (Germany), 01 - 05 Apr 2019

J. Neugebauer co-organized the Topical Session "Symposium "Big data analysis in Materials Science" at the DPG Spring Meeting 2019 in Regensburg (Germany), 01 Mar - 05 Apr 2019

B. Gault was the IFES (International Field Emission Society) Chair for "Microscopy & Microanalysis 2019" in Portland, OR (USA), 04 - 08 Aug 2019

G. Dehm co-organized the symposium "Metals, Alloys and Intermetallics" at the Microscopy Conference MC2019, Berlin (Germany), 01 - 05 Sep 2019

F. Körmann co-organized the workshop "Conference on Theory of Complex Disorder in Materials" at the Linköping University, Linköping, (Sweden), 16 - 18 Sep 2019

M. Palm and *F. Stein* co-organized the international conference "Intermetallics 2019" at the Educational Center Kloster Banz, Bad Staffelstein (Germany), 30 Sep - 04 Oct 2019

G. Dehm, *C. Liebscher*, *C. Scheu*, and *B. Völker* organized the „International Workshop on Advanced In Situ Microscopies of Functional Nanomaterials and Devices – IAMNano 2019" at MPIE, Düsseldorf (Germany), 27 - 30 Oct 2019

B. Gault, T. Schwarz, and L. Gomell co-organized the "NRW Atom probe user meeting" at the MPIE, Düsseldorf (Germany), 07 Nov 2019

M. Rohwerder organized the 9th GfKORR Work Group Meeting „Korrosionsuntersuchung und Überwachung" and the 8th GfKORR Work Group Meeting "Grundlagen und Simulation" at the MPIE, Düsseldorf (Germany), 21 Nov 2019

2020

S. Brinckmann, *G. Dehm*, and *C. Scheu* organized the Symposium "Mechanics Meets Energy 2020" in Lingen (Germany), 13 - 17 Jan 2020

G. Dehm, *C. Kirchlechner*, and *U. Hangen* organized the "Nanobrücken 2020: A Nanomechanical Testing Conference", Düsseldorf (Germany), 04 - 06 Feb 2020

F. Stein and *M.Palm* co-organized the "4th MSIT Winter School on Materials Chemistry" at Ringberg Castle, Kreuth (Germany), 16 - 20 Feb 2020

J. Janssen, S. Surendralal, J. Neugebauer, Y. Lysogorskiy, and *R. Drautz*, organized the pyiron Virtual Workshop "Workflows for atomistic simulations", Düsseldorf (Germany), 15 - 17 Apr 2020

M. Ashton, M. Todorova, J. Neugebauer, organized the Workshop „Electrically triggered reactions at interfaces – from ab initio to the multiscale", Schloss Ringberg (Germany), 26 – 29. Apr 2020. Cancelled due to COVID-19.

A. A. El-Zoka, co-chaired the sessions "L04-Metal Organic Frameworks" and "L04-Nanoporous Materials by Dealloying" at the 237th ECS Meeting within the 18th International Meeting on Chemical Sensors, Sensors, held online, 30 May - 03 Jun 2020

D. Raabe, *J. Neugebauer*, *M. Kühbach*, *C. Freysoldt*, *B. Gault*, and *C. Liebscher* organized the online „BiGmax Workshop 2020 on Big-Data-Driven Materials Science", held online, 15 - 16 Jun 2020

B. Gault, T. Schwarz, and L. Gomell co-organized the "NRW Atom probe user meeting" at the MPIE Düsseldorf (Germany), 23 June 2020

L. Abdellaoui organized the "1st Virtual Thermoelectric Conference 2020" (VCT 2020), 21 - 23 July 2020

C.Scheu co-organized the “International Seminar Series on the Microstructure of Materials”, held online, 10 Sep 2022

M.J. Duarte, *M. Herbig*, *J. Sietsma*, and *R. Petrov* organized the symposium “Material Response to Complex Mechanical and Chemical Loading” at the MSE 2020 conference, held online, 22 - 25 Sep 2020

M. Herbig organized the symposium “Material Response to Complex Mechanical and Chemical Loading” at the MSE 2020 conference, held online, 22 - 25 Sep 2020

L. Huber co-organized the symposium “Predicting Interface Structure and Dynamics” at the Materials Science and Engineering (MSE) Congress, held online, Darmstadt (Germany), 22 - 25 Sep 2020

C. Liebscher, *C. Kübel*, and *P. van Aken* organized the symposium ”Advanced Transmission Electron Microscopy for Materials Science“ at the MSE 2020 conference, held online, 22 - 25 September 2020

F. Stein and *M. Palm* organized the International Conference “TOFA 2020 - 17th Discussion Meeting on Thermodynamics of Alloys” at the Educational Center Kloster Banz, Bad Staffelstein (Germany), 28 Sept - 03 Oct 2020

T. Hickel, J. Neugebauer, R. Drautz, and *F. Soisson* organized the “Ab initio Description of Iron and Steel (ADIS2020)” Workshop “Diffusion and Precipitation”, Schloss Ringberg (Germany), held online, 02 – 05 Nov 2020

R. Otis, J. Janssen, T. Hickel, J. Neugebauer, B. Bocklund, and *Z.K.Liu* organized the Workshop “Software Tools from Atomistics to Phase Diagrams”, held online, 10 – 11 Nov 2020

2021

M. Todorova and *N. Hörmann*, organized the mini-Symposium “Electrified Solid - Liquid interfaces” at the Online Spring Meeting of the Surface Science Division of the German Physical Society (DPG), 01 – 04 Mar 2021

Y. Lysogorskiy, R. Drautz, S. Surendralal, J. Janßen, and *J. Neugebauer*, organized the Workshop “Workflows for Atomistic Simulations”, held online, 10 – 12 Mar 2021

S. Antonov co-organized the “Defect and Phase Transformation Pathway Engineering for Desired Microstructures” at TMS 2021 Annual Meeting, held online, 15 – 18 Mar 2021

G. Dehm, *C. Gammer, Sang Ho Oh*, and *K.Y. Xie* organized the symposium “In-situ mechanical testing of materials at small length scales, modelling and data analysis” at the 2021 MRS Virtual Spring Meeting, held online, 17 - 23 Apr 2021

B. Gault and *L. Gomell* co-organized the” NRW Atom probe user meeting” at the MPIE, Düsseldorf (Germany) 28 - 29 Apr 2021

M.J. Duarte, *M. Cordill*, and *M.T. Lin* organized the symposium “Mechanical Properties and Adhesion” at the 47th International Conference on Metallurgical Coatings & Thin Films (ICMCTF 2020), held online, 26 - 30 Apr 2021

F. Stein and *M.Palm* co-organized the “5th MSIT Winter School on Materials Chemistry”, held online, 02 - 06 May 2021

C. Freysoldt, *C. Liebscher*, and *R. Ernstorfer* organized the online BiGmax summer school “Harnessing big data in materials science from theory to experiment“, 13 - 17 Sep 2021

M. Palm and *F. Stein* co-organized the international conference “Intermetallics 2021” at the Educational Center Kloster Banz, Bad Staffelstein (Germany), 04 - 08 Oct 2021

M. Rohwerder co-organized the “C03 - Corrosion Mechanisms and Methods” symposium at the 240th Meeting of the ECS, held online, 10 - 14 Oct 2021

Institute colloquia and invited seminar talks

2019

Liang Gao, Max Planck Institute for Plasma Physics, Munich, Germany: Interaction of Deuterium Plasma with Tungsten: With Focus on Hydrogen-Induced Damage (03 Jan 2019)

Stefan Pogatscher, Montanuniversität Leoben, Leoben, Austria: Phase Transitions in Non-Equilibrium Metallic Systems (18 Jan 2019)

Andrea Brognara, Politecnico di Milano, Milan, Italy: Synthesis and Integration of Nanostructured TiO_2 Films with Plasmonic Au Nanoparticles (18 Jan 2019)

Felix Gunkel, Institute of Electronic Materials, RWTH Aachen University, Aachen, Germany and Peter Gruenberg Institut (PGI-7) – Forschungszentrum Jülich, Jülich, Germany: Nanoscale Thermodynamics at Complex Oxide Surfaces and Interfaces for Application in Electronics, Sensing, and Energy Conversion (31 Jan 2019)

Minoru Otani, National Institute of Advanced Industrial Science and Technology (AIST), Tsukuba, Japan: Theoretical Description of the Equilibrium Potential of an Electrochemical Reaction on a Metal Electrode (05 Feb 2019)

Timon Rabczuk, Bauhaus University Weimar, Weimar, Germany: Computational Modeling of Moving Boundary Problems (07 Feb 2019)

Bianca Maria Colosimo, Politecnico di Milano, Milan, Italy: In-situ Sensing and Monitoring of Metal Additive Manufacturing Processes (11 Feb 2019)

Jean-Sébastien Micha, European Synchrotron Radiation Facilities (CEA-INAC: CRG-IF at BM32), Grenoble, France: Facilities and Capabilities of French CRG-IF BM32 Beamline to Study Materials (11 Feb 2019)

Stéphane Berbenni, *Vincent Taupin*, Université de Lorraine, Metz, France: A Micromechanical Approach Based on Fourier Transforms and Continuum Dislocation Mechanics to Simulate Grain Size Effects in Polycrystals (18 Mar 2019)

Antoine Guitton, Université de Lorraine, Metz, France: Accurate Electron Channeling Contrast Imaging (aECCI): A Powerful Tool for Understanding the Fundamental Deformation Mechanisms of Materials (18 Mar 2019)

Sang Ho Oh, Sungkyunkwan University, Seoul, Republic of Korea: TEM Studies on Materials with a Negative Poisson's Ratio (21 Mar 2019)

Claudia Felser, Max Planck Institute for Chemical Physics of Solids, Dresden, Germany: The Heusler System (for Thermoelectric Application): How You Can Use the Periodic Table as a Lego Box to Build the States You Are Interested in (9 Apr 2019, Colloquium)

Henry Ovri, Helmholtz Zentrum Geesthacht, Geesthacht, Germany: Nanoindentation Based Investigations of PLC-type Plastic Instability (11 Apr 2019)

Gabi Schierning, Leibniz-Institut für Festkörper- und Werkstoffforschung Dresden, Dresden Germany: Making Quantum Transport Visible in Thermoelectric Bi_2Te_3 Nanoparticles (24 Apr 2019)

Ondrej L. Krivanek, Nion R&D, Kirkland, WA, and Arizona State University, Tempe, AZ, USA: Aberration-corrected STEM and Ultra-high Energy Resolution EELS (6 May 2019)

Thomas D. Swinburne, CNRS, CINaM, Marseille, France: Multiscale Materials Modelling using Sampling and Statistical Mechanics (7 May 2019)

Christian Greiner, Karlsruhe Institute of Technology (KIT), Karlsruhe, Germany: Deformation Mechanisms in Metals under a Tribological Load (16 May 2019)

Jakob Schwiedrzik, Empa - Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland: Micromechanics of Bone: Fundamental Research and Clinical Applications (17 May 2019)

Viet Anh Ha, Université catholique de Louvain (UCL), Louvain-la-Neuve, Belgium: High throughput computing for high mobility p-type transparent conducting materials (22 May 2019)

Natasha Stephens, Plymouth Electron Microscopy Centre, University of Plymouth, UK: Exploring the Solar System; From the Nano to Astronomical Scale (4 June 2019, Colloquium)

Aleksandar Zeradjanin, Universität Bremen, Germany: What is the trigger for hydrogen evolution reaction? (5 June 2019)

Ümit Güder, Çanakkale Onsekiz Mart University, Turkey: History of Iron Metallurgy in Anatolia (24 June 2019)

Ivan Kaban, Leibniz-Institut für Festkörper- und Werkstoffforschung Dresden, Dresden Germany: Experimental studies of phase formation and microstructure development upon non-equilibrium solidification (28 June 2019)

Moritz to Baben, GTT-Technologies, Aachen, Germany: The High-Throughput Way from 0 K to Relevant Temperatures to Enable the Circular Economy (2 July 2019)

Paul Coxon, University of Cambridge, Cambridge, UK: A Sustainable Molten Salt Route for the Electro-extraction and Electro-refining of Low-grade Ores to Yield High Purity Titanium Powders (4 July 2019, Colloquium)

Dominik Noeger, Montanuniversität Leoben, Leoben, Austria: Interaction of the H^2 molecule with carbon nanostructures: A DFT study (17 July 2019)

Yolita Eggeler, University of California, Santa Barbara, CF, USA: Using Analytical Electron Microscopy to Study Microstructural Evolution and Its Effect on Structural & Functional Properties (19 July 2019)

Björn Grüning, University of Freiburg, Germany: Integrative Data Analysis - Building Frameworks to Serve the 21th Century Data Science Problems (31 July 2019)

Hung-wei (Homer) Yen, National Taiwan University, Taipei, Taiwan: Machine Learning Enabled Materials Design: Low-Modulus Ti Alloys (22 Aug 2019)

Tobias Brink, EPFL - École Polytechnique Fédérale de Lausanne, Switzerland: Molecular Dynamics Simulations and Beyond for Plasticity and Wear of Metals (27 Aug 2019)

Erik Bitzek, Friedrich-Alexander-Universität Erlangen-Nürnberg (FAU), Erlangen, Germany: Towards a More Realistic Modeling of Defect-Defect Interactions in Atomistic Simulations of Crystalline Materials (5 Sept 2019)

Binglun Yin, École polytechnique fédérale de Lausanne, Lausanne, Switzerland: Yield Strength and Misfit Volumes of NiCoCr and Implications for Short-Range-Order (18 Sept 2019)

Taher Saif, University of Illinois at Urbana-Champaign, IL, USA: Lessons Learned from Nano Scale Specimens Tested by MEMS Based Apparatus (24 Sept 2019)

John Wheeler, University of Liverpool, UK: Interactions of Stress with Chemical Processes in Crystalline Materials (26 Sept 2019, Colloquium)

Zi-Kui Liu, Pennsylvania State University, State College, PA, USA: Where Do Thermodynamics and Transport Kinetics Meet? (27 Sept 2019)

Uwe Glatzel, University of Bayreuth, Bayreuth, Germany: Close Packed Phases in Nickel-Based Superalloys - Investigation by Diffusion Multiples (30 Sept 2019)

Mehmet Acet, University of Duisburg-Essen, Duisburg, Germany: Inducing Strong Magnetism in High-Entropy Alloys by Exploiting Their Anti-Invar Properties (8 Oct 2019)

Dietmar Hömberg, WIAS & Institut für Mathematik, TU Berlin, Germany: Mathematics for Steel Production and Manufacturing (14 Oct 2019)

Benjamin Balke, Fraunhofer Research Institution for Materials Recycling and Resource Strategies IWKS, Hanau, Germany: Designing Thermoelectric Highly Efficient Hf-Free n- & p-type Heusler Compounds via Phase Separation and Nano-Composites (17 Oct 2019)

James P. Best, RWTH University Aachen: Multi-Scale Design and Analyses of Advanced Materials: Experimental Approaches (17 Oct 2019)

Peter Schweizer, Friedrich-Alexander-Universität Erlangen-Nürnberg (FAU), Erlangen, Germany: Manipulation of Individual Defects in 2D and Layered Materials (18 Oct 2019)

Rajaprakash Ramachandramoorthy, EMPA, Thun, Switzerland: Pushing the Boundaries of Micro and Nanomechanics (22 Oct 2019)

Nesma Aboulkhair, Centre for Additive Manufacturing (CfAM), University of Nottingham, Nottingham, UK: An Approach to Powder Feedstock Modification for *in-situ* Alloying in Metal Additive Manufacture to Improve the Processability of Difficult-to-Process Materials using Carbon Nanotubes (31 Oct 2019)

Marco Simonelli, Centre for Additive Manufacturing (CfAM), University of Nottingham, Nottingham, UK: Development of Novel Titanium Alloys for Laser Powder Bed Fusion (31 Oct 2019)

Masaaki Sugiyama, Osaka University, Japan: Use of Focused Ion Beam (FIB) Instruments for TEM Sample Preparation and Standardization of FIB Processes (07 Nov 2019)

Michael Farle, University of Duisburg-Essen, Duisburg: Novel Magnetic Functionalities in Classic Materials (11 Nov 2019, Colloquium)

Jürgen Bär, Bundeswehr University Munich: Investigation of Thermal Effects in Cyclic Loaded Metallic Materials (20 Nov 2019)

Marvin Poul, University of Stuttgart, Stuttgart, Germany: From Atom Probe Tomography to CALPHAD modeling: Estimating T_c from local concentration fluctuations (26 Nov 2019)

Szilvia Kalácska, Empa Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland: Characterization of Plastically Deformed Metals by 3D High Resolution Electron Backscatter Diffraction (HR-EBSD) at the Micron Scale (11 Dec 2019)

2020

Michael W. Finnis, Imperial College London, London, UK: Interactions of H with vacancies in Fe (15 Jan 2020)

Stefan Sandfeld, TU Bergakademie Freiberg, Freiberg, Germany: Software solutions for micromechanical materials modeling (16 Jan 2020)

Nima H. Siboni, Technische Universität Berlin, Berlin, Germany: Non-Monotonic Rheology of a Magnetic Liquid Crystal System in an External Field (16 Jan 2020)

Christian Silbermann, Technische Universität Chemnitz, Chemnitz, Germany: On the modeling of dislocation- and deformation-induced plastic localization phenomena of metallic materials (28 Jan 2020)

Motomichi Koyama, Tohoku University, Sendai, Japan: Effects of Dislocation Planarity and Compositional Complexity on Hydrogen Embrittlement of Austenitic Steels (17 Feb 2020)

Louis Vanduyfhuys, University of Ghent, Ghent, Belgium: Unraveling the thermodynamic conditions for negative gas adsorption in soft porous crystals (17 Feb 2020)

Erik Bitzek, Friedrich-Alexander-Universität Erlangen-Nürnberg (FAU), Erlangen: Influence of Deformation-Induced Topological Anisotropy on Mechanical Properties of Silica Glass: An Atomistic Study (18 Feb 2020)

Jeffrey Snyder, Northwestern University, Evanston, IL, USA: Engineering Defects and Grain Boundaries in Thermoelectric Materials (21 Feb 2020, Colloquium)

Yang Bai, Technical University Darmstadt, Darmstadt, Germany: Chemo-Mechanical Modeling of the Diffusion and Fracture in Cathode Materials of Lithium-Ion Batteries (27 Feb 2020)

Güven Kurtuldu, ETH Zurich, Zurich, Switzerland: Quasicrystal-Induced Nucleation Mechanism in Undercooled Liquids (14 July 2020)

Hortense Le Ferrand, Nanyang Technological University, Singapore: Bioinspired Multifunctional Structural Materials (13 Aug 2020)

Ralf Drautz, ICAMS, Bochum, Germany: Atomic cluster expansion for accurate and transferable interatomic potentials (24 Aug 2020)

Megan Cordill, Eric Schmid Institute of Materials Science, Leoben, Austria: Evaluating Electro-Mechanical Reliability using *in-situ* Methods (27 Aug 2020)

Gaurav Mohanty, Tampere University, Tampere, Finland: Reliable Extraction of Deformation Activation Parameters from Transient and High Strain Rate Micromechanical Tests (10 Sept 2020)

Stefan Pauliuk, University of Freiburg, Freiburg, Germany: Sustainability Challenges in the Global Steel Cycle (21 Oct 2020)

Jean-Philippe Couzinié, Université Paris Est, Paris, France: Insights into the Exploration of Refractory High-Entropy Alloys: From the Design to the Deformation Mechanisms of these Complex Materials (6 Nov 2020)

Vanessa Wood, ETH Zurich, Zurich, Switzerland: Volumetric Imaging for Better Batteries (19 Nov 2020, Colloquium)

Samuel Forest, Mines ParisTech, Paris, France: A Cosserat crystal plasticity and phase field approach to grain boundary migration and recrystallization (1 Dec 2020, Colloquium)

Maurine Montagnat, Institute of Geosciences of Environment, CNRS - University of Grenoble Alpes, and Centre d'Etude de la Neige, Centre National de Recherches Météorologiques – Météo-France, CNRS, Grenoble, France: Inside a Deep Ice Core. From Small Scale Processes to Large Scale Flow (2 Dec 2020)

Atsushi Togo, National Institute for Materials Science (NIMS), Tsukuba, Japan: Tool development for phonon calculations and applications (2 Dec 2020)

Norbert Kruse, Washington State University, Pullman, WA, USA: Imaging and Chemical Probing of Catalytical Reactions with Subnanometer Resolution (7 Dec 2020)

2021

Alán Aspuru-Guzik, University of Toronto, Toronto, Canada: The Materials for Tomorrow, Today (11 Jan 2021, Colloquium)

Hosni Idrissi, Ecole Polytechnique de Louvain, Louvain-la-Neuve, Belgium: Stress Induced Grain Boundary Processes in Metals and Minerals: New Insights from *in-situ* TEM Nanomechanical Testing (14 Jan 2021)

Roland Verreet, Wire Rope Technology, Aachen, Germany: Wire Ropes and Sustainability. An Overview by the Rope Pope! (14 Jan 2021)

Markus Stricker, ICAMS, Bochum, Germany: Prismatic Slip in Magnesium (18 Jan 2021)

Yansong Shen, University of New South Wales, Sydney, Australia: Process Modeling of Reacting Flows and Industry Applications: Recent Work of Hydrogen Injection Processes in Ironmaking Blast Furnaces (21 Jan 2021)

Ben Britton, Imperial College London, UK: No Sexuality Please, We're Scientists (22 Jan 2021)

Emilio Martínez-Pañeda, Imperial College London, London, UK: Predictive Modelling of Hydrogen Assisted Fracture (18 Feb 2021)

Benoit Merle, Friedrich-Alexander-Universität Erlangen-Nürnberg (FAU), Erlangen, Germany: Nanoindentation at High Strain Rates: Challenges and Recent Advances (23 Feb 2021)

Johannes Schenk, Montanuniversität Leoben, Leoben, Austria: Hydrogen Meets Future Steelmaking - Applied Methods and Achieved Results for the Investigation of the Kinetics of Iron Ore Reduction with Hydrogen at Montanuniversität Leoben (24 Feb 2021, Colloquium)

Yong Zhu, North Carolina State University, Raleigh, NC, USA: MEMS-based *in-situ* Nanomechanics of Crystalline Nanowires (4 Mar 2021)

Bai-Xiang Xu, Technical University of Darmstadt, Darmstadt, Germany: Multiphysics Phase-Field Modeling and Simulation of Advanced Materials and Processing (5 Mar 2021)

Di Wan, Norwegian University of Science and Technology (NTNU), Trondheim, Norway: Hydrogen Effects on Metallic Materials Studied via Small-Scale *in-situ* Techniques (18 Mar 2021)

Shirley Meng, University of California San Diego, CF, USA: From Atom to System - How to Enable the Tera-Scale Energy Transition (25 Mar 2021, Colloquium)

Kathrin Greiff, RWTH Aachen University, Aachen, Germany: More than Recycling – Challenges & Potentials of the Circular Economy in the Case of Metals (29 Mar 2021, Colloquium)

Sho Hayakawa, University of Tennessee, Knoxville, Tennessee, USA: Active learning and on-the-fly KMC (30 Mar 2021)

Lukas Stemper, Montanuniversität Leoben, Leoben, Austria: Crossover Alloys – A New Approach for Future Aluminum Alloys (7 Apr 2021)

Fabrice Patisson, Mines Nancy, University of Lorraine, Nancy, France: Hydrogen Ironmaking: Kinetics and Modeling of the Iron Ore Reduction (8 Apr 2021)

Walid Hetaba, Max Planck Institute for Chemical Energy Conversion, Mülheim an der Ruhr: ChemiTEM – An Easy to Use TEM for Chemistry and Material Science (19 Apr 2021)

Varatharaja Nallathambi, Indian Institute of Technology Madras, Chennai, India: Phase Stability Studies and Evaluation of Thermal & Mechanical Properties of Entropy Stabilized Transition Metal Oxides (21 Apr 2021)

Balila Nagamani Jaya, Indian Institute of Technology (IIT) Bombay, India: Design and Applications of Length Scale Compatible Fracture Test Geometries (10 May 2021)

Maria Ibáñez, Institute of Science and Technology (IST) of Austria, Klosterneuburg, Austria: Solution-Processed Thermoelectric Materials: The Case of SnSe (10 May 2021)

Maryam Ghazisaeidi, Ohio State University, Columbus, Ohio, USA: Multi-cell Monte Carlo Method for Phase prediction (12 Mai 2021)

Rebecca Janisch, ICAMS, Bochum, Germany: Hydrogen Enhanced Decohesion at Grain Boundaries - Insights from ab-initio Calculations (27 May 2021)

Alejandro A. Franco, Université de Picardie Jules Verne, Amiens, France: Digitalization of Battery Manufacturing through Artificial Intelligence and Multiscale Modeling (01 June 2021)

Napat Vajragupta, ICAMS, Bochum, Germany: Micromechanics of Large Deformations (10 June 2021)

Rainer Abart, University of Vienna, Vienna, Austria: Growth of Mg-Aluminate Spinel at MgO-Al_2O_3 Contacts: Experiment, Nature, and Some Theory (17 June 2021)

Fadi Aldakheel, Leibniz University Hannover, Hannover, Germany: Global-Local Techniques for Adaptive Phase-Field Fracture (17 June 2021)

Philip de Goey, Eindhoven University of Technology, eindhoven, The Netherlands: Metal Energy Carriers: Renewable Fuels of the Future (30 June 2021)

Ramin Bostanabad, University of California, Irvine, CA, USA: Artificial Intelligence for Engineering Design and Computational Mechanics (6 July 2021)

Daniel Şopu, Erich Schmid Institute of Materials Science, Leoben, Austria: STZ Vortex Unit – The Key to Understand and Control Shear Banding in Metallic Glasses (6 July 2021)

Veronica Augustyn, North Carolina State University, Raleigh, NC, USA: Electrochemical Capacitance under Confinement: Implications for Electrochemical Energy Storage and Conversion (14 July 2021)

Olga Kasian, Helmholtz Zentrum Berlin, Berlin, Germany: Electrochemistry in the Renewable Energy Cycle (27 July 2021)

Leopold Lahn, Helmholtz Zentrum Berlin, Berlin, Germany: Ir based alloys for energy conversion applications (27 July 2021)

Marc Ledendecker, TH Darmstadt, Darmstadt, Germany: Design Strategies for Electrocatalyst Materials for the Oxygen Reduction Reaction (27 July 2021)

Ferdinand Biere, DeepMetis, Berlin, Germany: Be Your Own Boss – How to Found a Start Up (16 Aug 2021)

Yujiao Li, Ruhr-Universität Bochum, Bochum, Germany: Accelerated Atomic-Scale Exploration of Phase Evolution in High-Entropy Alloys (17 Aug 2021)

Fabian Sewerin, Otto von Guericke University Magdeburg, Magdeburg, Germany: Modelling the Combustion of Metal Powders in Laminar and Turbulent Flames (20 Aug 2021)

Sha Liu, IMDEA Materials Institute, Madrid, Spain: First principles prediction of Al and Mg alloys phase diagrams (24 Aug 2021)

Sergei V. Kalinin, Center for Nanophase Materials Sciences, Oak Ridge National Laboratory, Oak Ridge, TN, USA: Realizing Physical Discovery in Imaging with Machine Learning (2 Sep 2021)

Eugen Rabkin, Department of Materials Science and Engineering, Technion, Haifa, Israel: Exploring the limits of metal strength (13 Sep 2021)

Nidhin George Mathews, Metallurgical Engineering and Materials Science Department, Indian Institute of Technology Bombay, India: Guidelines on how best to do a micro-cantilever bending based fracture test (14 Sep 2021)

Rossitza Pentcheva, Faculty of Physics, Theoretical Physics, University of Duisburg-Essen: Understanding and Improving the Catalytic Activity of Transition Metal Oxide Surfaces: Insights from DFT+U Calculations (23 Sep 2021)

Robert E. Sanders, Chongqing University, Chongqing, People's Republic of China: The Sustainability Challenge for the Aluminum Industry (30 Sep 2021)

Torsten Brezesinski, Institute of Nanotechnology, Karlsruhe Institute of Technology (KIT), Eggenstein-Leopoldshafen, Germany: Tailoring Layered Ni-rich Oxide Cathode Materials for Solid-State Battery Applications (1 Oct 2021)

Guillaume Stechmann, ArcelorMittal Global R&D - Steelmaking Process, Maizières-les-Metz, France: Machine Learning for the Steel Industry: Behind the Buzzword (5 Oct 2021)

Robert E. Sanders, Chongqing University, Chongqing, People's Republic of China: Aluminum Usage for Light Vehicles and EV's Evolution and Challenges (7 Oct 2021)

Frank Renner, Hasselt University, Belgium: Looking Deep into Li-Ion Batteries: Advanced Characterization for New Technologies (11 Oct 2021)

Robert E. Sanders, Chongqing University, Chongqing, People's Republic of China: Aluminum Alloy Aircraft Products: Performance and Manufacturing Considerations (21 Oct 2021)

Katayun Barmak, Columbia University, New York, USA: Towards a Predictive Theory of Grain Growth: Experiments and Simulations (25 Oct 2021)

Jafar Safarian, Norwegian University of Science and Technology (NTNU), Trondheim, Norway: Silicon Purification through Metallurgical Processes for PV Silicon Production (29 Oct 2021)

Andras Kovacs, Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons, Forschungszentrum Jülich: What Can We Learn from Magnetic Imaging in TEM? (4 Nov 2021)

Jeffrey Bergthorson, McGill University, Montreal, Canada: Metal Fuels for Zero-Carbon Heat and Power (8 Nov 2021)

William Chueh, Stanford University, CA, USA: Designing a More Homogenous Battery: Emergent Electrochemical Phenomena at the Mesoscale (9 Nov 2021)

Katie L. Moore, University of Manchester, UK: Localisation of Hydrogen and Deuterium in Metallurgical Samples with NanoSIMS (10 Nov 2021)

Wolfgang Zeier, University of Münster & Helmholtz-Institute Münster, Germany: Understanding (Effective) Ionic Transport in Solids and Solid-state Batteries (11 Nov 2021)

Gunther Richter, Max Planck Institute for Intelligent Systems, Stuttgart: In situ Transmission Electron Microscopy Observations of Co Nanowhiskers (11 Nov 2021)

Christopher Aiden-Lee Jackson, University of Manchester, UK: What Does Race Have to Do with Science (1 Dec 2021)

Lectures and teaching at universities

2018 (not included in the Scientific Report 2016 - 2018)

Dehm, G. Transmissionselektronenmikroskopie für Fortgeschrittene. Ruhr-Universität Bochum, WS 2018/2019

Dehm, G.; Scheu, C. Atomare Charakterisierung von Werkstoffen. Heinrich-Heine Universität Düsseldorf, WS 2018/2019

Liebscher, C. Transmissionselektronenmikroskopie für Fortgeschrittene (Übung). Ruhr-Universität Bochum, WS 2018/2019

Roters, F. Prozess- und Werkstoffsimulation. RWTH Aachen, WS 2018/2019

Scheu, C. Electron Microscopy and Analytical Techniques. RWTH Aachen, WS 2018/2019

2019

Dehm, G. Mechanische Eigenschaften in kleinen Dimensionen. Ruhr-Universität Bochum, SS 2019

Dehm, G. Transmissionselektronenmikroskopie für Fortgeschrittene. Ruhr-Universität Bochum, WS 2019/2020

Hickel, T. Introduction to Quantum Mechanics in Solid-State Physics. Ruhr-Universität Bochum, WS 2019/2020

Liebscher, C. Transmissionselektronenmikroskopie für Fortgeschrittene (Übung). Ruhr-Universität Bochum, WS 2019/2020

Neugebauer, J. Application and Implementation of Electronic Structure Methods. Ruhr-Universität Bochum, WS 2019/2020

Raabe, D. Micromechanics of Materials. RWTH Aachen, SS 2019

Rohwerder, M. Surface Science and Corrosion. Ruhr-Universität Bochum, WS 2019/2020

Roters, F. Prozess- und Werkstoffsimulation. RWTH Aachen, WS 2019/2020

Scheu, C. Electron Microscopy and Analytical Techniques. RWTH Aachen, WS 2019/2020

Scheu, C. Moderne Material- und Werkstoffcharakterisierung: Vom Atom zum Bauteil. RWTH Aachen, SS 2019

Zaefferer, S. Lehrgang EBSD: Rückstreuelektronenbeugung (EBSD) im REM. Fachhochschulzentrum (FHZ) Münster, March 2019

Zaefferer, S.; Hickel, T.; Song, W. Microstructures, Microscopy and Modelling. RWTH Aachen, SS 2019

2020

Dehm, G. Mechanische Eigenschaften in kleinen Dimensionen. Ruhr-Universität Bochum, SS 2020

Dehm, G. Transmissionselektronenmikroskopie für Fortgeschrittene. Ruhr-Universität Bochum, WS 2020/2021

Dehm, G.; Scheu, C. Atomare Charakterisierung von Werkstoffen. Heinrich-Heine Universität Düsseldorf, WS 2020/2021

Hickel, T. Introduction to Quantum Mechanics in Solid-State Physics. Ruhr-Universität Bochum, WS 2020/2021

Liebscher, C. Transmissionselektronenmikroskopie für Fortgeschrittene (Übung). Ruhr-Universität Bochum, WS 2020/2021

Neugebauer, J. Application and Implementation of Electronic Structure Methods. Ruhr-Universität Bochum, WS 2020/2021

Neugebauer, J. Electronic Structure Methods. Ruhr-Universität Bochum, SurMat T3 Module Simulations and Modelling Lecture, WS 2020/2021

Raabe, D. Micromechanics of Materials. RWTH Aachen, SS 2020

Raabe, D. Sustainable Materials Science and Green Metallurgy. RWTH Aachen, SS 2020

Rohwerder, M. IMPRS-SurMat Lecture: Corrosion. University of Duisburg-Essen and Ruhr-Universität Bochum, SS 2020

Rohwerder, M. Surface Science and Corrosion. Ruhr-Universität Bochum, WS 2020/2021

Roters, F. Prozess- und Werkstoffsimulation. RWTH Aachen, WS 2020/2021

Scheu, C. Electron Microscopy and Analytical Techniques. RWTH Aachen, WS 2020/2021

Scheu, C. Moderne Material- und Werkstoffcharakterisierung: Vom Atom zum Bauteil. RWTH Aachen, SS 2020

Springer, H. Allgemeine Werkstofftechnik. RWTH Aachen, WS 2020/2021

Zaefferer, S. Fundamentals and practical aspects of texture and microstructure measurements using EBSD-based orientation microscopy and related techniques. IIT Madras, India, Jan 2020

Zaefferer, S. Fundamentals and practical aspects of texture and microstructure measurements using EBSD-based orientation microscopy and related techniques. MPIE Düsseldorf, Feb and Mar 2020

Zaefferer, S.; Hickel, T.; Song, W. Microstructures, Microscopy and Modelling. RWTH Aachen, SS 2020

2021

Dehm, G. Mechanische Eigenschaften in kleinen Dimensionen. Ruhr-Universität Bochum, SS 2021

Dehm, G. Transmissionselektronenmikroskopie für Fortgeschrittene. Ruhr-Universität Bochum, WS 2021/2022

El-Zoka, A. Introduction to Atom Probe Tomography. MPIE Düsseldorf, IMPRS-SurMat, Aug 2021

Gault, B. Graduate course on Atom Probe Tomography, as part of the Centre for Doctoral Training on Materials Characterisa-tion. Imperial College London, UK, SS 2021

Liebscher, C. Transmissionselektronenmikroskopie für Fortgeschrittene (Übung). Ruhr-Universität Bochum, WS 2021/2022

Raabe, D.; Cojocaru-Mirédin, O.; El-Zoka, A.; Kontis, P.; Ma, Y.; Mianroodi, J. R.; Ponge, D.; Souza Filho, I. R.; Sandlöbes-Haut, S.; Zaefferer, S.; Zhao, H. Sustainable Materials Science and Green Metallurgy (Sustainable Materials and Metallurgical Science & Engineering). RWTH Aachen, SS 2021

Rohwerder, M. Surface Science and Corrosion. Ruhr-Universität Bochum, WS 2021/2022

Scheu, C. Advanced Characterisation. RWTH Aachen, WS 2021/2022

Scheu, C. Moderne Material- und Werkstoffcharakterisierung: Vom Atom zum Bauteil. RWTH Aachen, SS 2021

Springer, H. Allgemeine Werkstofftechnik. RWTH Aachen, WS 2021/2022

Zaefferer, S.; Hickel, T.; Song, W. Microstructures, Microscopy and Modelling. RWTH Aachen, SS 2021

Zaefferer, S.; Motaman, S. A. H. Metallic Materials (Microstructure, Microscopy, Modelling). RWTH Aachen, SS 2021

Invited talks at conferences and colloquia

2018 (not included in Scientific Report 2016 - 2018)

Dehm, G.: *Defects in alloys: New insights by advanced in-situ electron microscopy* (Symposium Netzwerk Elektronenmikroskopie: Struktur und Chemie an Werkstoffen und biologischen Systemen, Naturwissenschaftliches und Medizinisches Institut an der Universität Tübingen. Reutlingen, Germany. 12 Nov. 2018)

Garzón-Manjón, A.: *Controlling the structure and composition of multinary alloy nanoparticles for energy applications* (Leitat Institut Colloquium. Barcelona, Spain. 30 Nov. 2018)

Kürnsteiner, P.; Wilms, M. B.; Weisheit, A.; Bajaj, P.; Li, X.; Leinenbach, C.; Jägle, E. A.; Raabe, D.: *Process and Alloy Design for In-Situ Precipitation Strengthening of Al–Sc Alloys During Laser Metal Deposition* (Metal Additive Manufacturing Conference. Vienna, Austria. 21 Nov. 2018)

Kürnsteiner, P.; Wilms, M. B.; Weisheit, A.; Barriobero-Vila, P.; Jägle, E. A.; Raabe, D.: *Designing a novel Fe–Ni–Al–Ti Maraging steel tailor-made for Laser Metal Deposition* (Metal Additive Manufacturing Conference. Vienna, Austria. 21 Nov. 2018)

Kürnsteiner, P.; Wilms, M. B.; Weisheit, A.; Barriobero-Vila, P.; Jägle, E. A.; Raabe, D.: *Designing Fe–Ni–Al and Fe–Ni–Ti Maraging steels tailor-made for Laser Metal Deposition by exploiting Intrinsic Heat Treatment* (Alloys for Additive Manufacturing Symposium. Sheffield, UK. 03 Sep. 2018)

Li, Z.; Su, J.; Lu, W.; Wang, Z.; Raabe, D.: *Metastable high-entropy alloys: design, structure and properties* (2nd International Conference on High-Entropy Materials (ICHEM 2018). Jeju, South Korea. 09 Dec. 2018)

Liebscher, C.; Peter, N. J.; Meiners, T.; Frolov, T.; Dehm, G.: *Atomic scale observations of grain boundary phase transformations* (Materials Science and Engineering Congress 2018. Darmstadt, Germany. 26 Sep. 2018)

Raabe, D.; Kwiatkowski da Silva, A.; Ponge, D.; Li, Z.; Makineni, S. K.; Li, L.; Gault, B.: *Grain boundary segregation and transformation in complex alloys* (Fall Meeting. Boston, MA, USA. 25 Nov. 2018)

Raabe, D.; Lu, W.; Ponge, D.; Li, Z.; Kwiatkowski da Silva, A.; Makineni, S. K.; Kontis, P.; Wu, X.; Gault, B.: *Local and global alloy design: metastability of single lattice defects and alloys with extreme metastability* (MIT Workshop on Alloy Design. Cambridge, MA, USA. 30 Nov. 2018)

Raabe, D.; Ponge, D.; Kwiatkowski da Silva, A.; Makineni, S. K.; Peng, Z.; Li, L.; Kontis, P.; Wu, X.; Springer, H.; Belde, M. M.; Gault, B.: *Segregation and Transformation at Lattice Defects in Complex Alloys: A Microstructure Design Toolbox* (MRS Fall Meeting. Boston, MA, USA. 25 Nov. 2018)

Scheu, C.; Hengge, K. A.: *Unravelling catalyst growth and degradation mechanisms via STEM* (International Workshop on Advanced and In-situ Microscopies of Functional Nanomaterials and Devices, IAMNano 2018. Hamburg, Germany. 14 Oct. 2018)

Seol, J. B.; Ko, W.-S.; Bae, J. W.; Jo, Y. H.; Li, Z.; Choi, P.-P.; Raabe, D.; Kim, H. S.: *Transition in boron boundary cohesion from effectiveness to harmfulness with respect to application temperatures: high-entropy alloys and Ni-based superalloys* (2nd International Conference on High-Entropy Materials (ICHEM 2018). Jeju, South Korea. 09 Dec. 2018)

Stephenson, L.: *Advanced quantitative analysis for Atom Probe Tomography* (APT school at Groupe de Physique des Matériaux (GPM), Université de Rouen. Saint Etienne du Rouvray, France. 18 Oct. 2018)

Stephenson, L.; Rusitzka, A. K.; Gault, B.: *Seeing atoms in biological materials: a new frontier for atomic-scale tomography* (Volkswagen Stiftung Symposium. Bremen, Germany. 31 Oct. 2018)

Sun, B.; Ponge, D.; Fazeli, F.; Scott, C.; Yue, S.; Raabe, D.: *Revealing fracture mechanisms of medium manganese steels with and without delta-ferrite* (6th International Conference on Advanced Steels (ICAS 2018). Jeju, South Korea. 18 Nov. 2018)

Todorova, M.; Yoo, S.-H.; Surendralal, S.; Neugebauer, J.: *Ab initio modelling of solid/liquid interfaces: Challenges and insights* (DESY Seminar. Hamburg, Germany. 20 Oct. 2018)

Zaefferer, S.: *The importance of microstructures for the energy conversion efficiency of materials for photovoltaic and photothermic applications* (Development of Photovoltaic Solar Energy in Africa by the Year 2030. Abidjan, Republik Côte d'Ivoire. 08 Nov. 2018)

2019

Abdellaoui, L.; Zhang, S.; Rodenkirchen, C.; Zaefferer, S.; Bueno Villoro, R.; Guen, E.; Chapuis, P.-O.; Gomès, S.; Cojocaru-Mirédin, O.; Yu, Y.; Amouyal, Y.; Raabe, D.; Snyder, G. J.; Scheu, C.: *Correlation of Microstructures and Thermal Conductivity of the Thermoelectric Material* $Ag_{16.7}Sb_{30}Te_{53.3}$ (North American Thermoelectric workshop, Northwestern University. Eanston, Chicago, IL, USA. 30 Aug. 2019)

Abdellaoui, L.; Zhang, S.; Rodenkirchen, C.; Zaefferer, S.; Bueno Villoro, R.; Guen, E.; Chapuis, P.-O.; Gomès, S.; Cojocaru-Mirédin, O.; Yu, Y.; Amouyal, Y.; Raabe, D.; Snyder, G. J.; Scheu, C.: *Effect of planar defects on the thermal conductivity of* $Ag_{16.7}Sb_{30}Te_{53.3}$ *bulk thermoelectric* (6th German/Korean Thermoelectric workshop. Daegu, South Korea. 05 Jul. 2019)

Brinckmann, S.; Dehm, G.: *Severe deformation of a lamellar microstructure: pearlitic steel as a case study* (TMS 2019. San Antonio, TX, USA. 10 Mar. 2019)

Dehm, G.: *Do we understand the microstructure and properties of materials: New insights by advanced microscopy techniques* (Metallurgical Engineering and Materials Science Department, Indian Institute of Technology. Mumbai, India. 15 Jul. 2019)

Dehm, G.: *Können hohe Festigkeit und Zähigkeit in Verschleißschutzschichten kombiniert werden? Eine grundlegende Unter-suchung an* Mo_2BC (12. Tagung Gefüge und Bruch. Bochum, Germany. 13 Feb. 2019)

Dehm, G.: *Können hohe Festigkeit und Zähigkeit in Verschleißschutzschichten kombiniert werden? Eine grundlegende Untersuchung an* Mo_2BC (12. Tagung Gefüge und Bruch. Bochum, Germany. 13 Feb. 2019)

Dehm, G.: *Micro- and Nanomechanical Testing of Materials - From Materials Physics to Materials Design* (Convegno Nazionale INSTM XII. Ischia Porto, Italy. 21 Jul. 2019)

Dehm, G.: *Probing the mechanics of dislocation - grain boundary interactions: Lessons learned from in situ microcompression experiments* (14th International Conference on Local Mechanical Properties 2019 (plenary). Prague, Czech Republic. 06 Nov. 2019)

Dehm, G.: *Resolving grain boundary phase transformations by advanced STEM for fcc metals and multinary alloys* (6th International Symposium on Metastable, Amorphous and Nanostructured Materials (ISMANAM-2019). Chennai, India. 08 Jul. 2019)

Dehm, G.: *Towards understanding dislocation-based plasticity in high entropy alloys by in-situ TEM* (Spring Meeting of the German Physical Society (DPG). Regensburg, Germany. 01 Apr. 2019)

Diehl, M.; Cereceda, D.; Marian, J.; Liu, C.; Dong, J.; Wicke, M.; Brückner-Foit, A.; Kamachali, R. D.; Shanthraj, P.; Roters, F.; Eisenlohr, P.; Raabe, D.: *Coupling Crystal Plasticity and Phase Field Methods - The Future of Integrated Computational Materials Engineering?* (Recent Advances in the Modeling and Simulation of the Mechanics of Nanoscale Materials. Philadelphia, PA, USA. 23 Aug. 2019)

Diehl, M.; Cereceda, D.; Marian, J.; Liu, C.; Dong, J.; Wicke, M.; Brückner-Foit, A.; Kamachali, R. D.; Shanthraj, P.; Roters, F.; Eisenlohr, P.; Raabe, D.: *DAMASK: The Düsseldorf Advanced Materials Simulation Kit* (Seminar of the Department of Materials Science and Engineering of the Ohio State University. Columbus, OH, USA. 26 Aug. 2019)

Diehl, M.; Kühbach, M.; Kertsch, L.; Traka, K.; Raabe, D.: *Coupled Experimental–Computational Analysis of Primary Static Recrystallization in Low Carbon Steel* (Seminar of the Department of Mechanical Science and Engineering of the University of Illinois. Urbana-Champaign, Il, USA. 27 Sep. 2019)

Diehl, M.; Shanthraj, P.; Eisenlohr, P.; Roters, F.; Raabe, D.: *Beyond Crystal Plasticity – Developing Tools for Integrated Computational Materials Engineering* (Seminar of the Tasan Group, MIT. Boston, MA, USA. 19 Jul. 2019)

Duarte, M. J.; Fang, X.; Rao, J.; Brinckmann, S.; Dehm, G.: *Hydrogen-metal interactions by in-situ and ex-situ nanoindentation* (2nd LINCET Symposium, Downing College. Cambridge, UK. 05 Apr. 2019)

El-Zoka, A.; Langelier, B.; Newman, R. C.: *Characterization and Functional Improvement of Nanoporous Metals* (236th ECS Meeting. Atlanta, GA, USA. 13 Oct. 2019)

Freysoldt, C.: *Concepts and algorithms in SPHInX (Weierstraß-Institut für Angewandte Analysis und Stochastik, Online Seminar*. Berlin, Germany. 09 May 2019)

Freysoldt, C.: *Concepts and algorithms in SPHInX* (Weierstraß-Institut für Angewandte Analysis und Stochastik, Invited Seminar. Berlin, Germany. 09 May 2019)

Freysoldt, C.: *Modelling of charged point defects with density-functional theory* (4th International Workshop on Models and Data for Plasma-Material Interaction in Fusion Devices, National Institute for Fusion Science (NIFS). Toki, Japan. 18 Jun. 2019)

Garzón-Manjón, A.; Meyer, H.; Grochla, D.; Löffler, T.; Savan, A.; Schuhmann, W.; Ludwig, A.; Scheu, C.: *Pathways for fabrication of amorphous and crystalline multinary nanoparticles for electrocatalyst* (14th International Conference on Materials Chemistry MC14. Birmingham, UK. 08 Jul. 2019)

Gault, B.: *A not-so-brief introduction to atom probe tomography: from fundamentals to atomic-scale insights into engineering materials* (Seminar, Imperial College London. London, UK. 19 Feb. 2019)

Gault, B.: *An introduction to atom probe tomography: from fundamentals to atomic-scale insights into engineering materials* (Seminar, University of British Columbia. Vancouver, BC, Canada. 24 May 2019)

Gault, B.: *An introduction to atom probe tomography: from fundamentals to atomic-scale insights into engineering materials* (Seminar, University of Manchester. Manchester, UK. 24 Jun. 2019)

Gault, B.: *An introduction to atom probe tomography: from fundamentals to atomic-scale insights into engineering materials* (Rolls Royce Lunch Time Seminar. Derby, UK. 01 Jul. 2019)

Gault, B.: *Atom Probe Tomography to help Understand Deformation Mechanisms in Metallic Alloys* (The International Conference on Metallurgical Coatings and Thin Films 2019. San Diego, CA, USA. 19 May 2019)

Gault, B.: *Can machine learning bring atom probe microscopy closer to analytical atomic-scale tomography* (12th International Symposium on Atomic Level Characterizations for New Materials and Devices (ALC 19). Kyoto, Japan. 20 Oct. 2019)

Gault, B.: *Quantifying hydrogen by atom probe tomography* (Seminar, Oxford University. Oxford, UK. 25 Jun. 2019)

Gault, B.: *Some kind of introduction to atom probe tomography* (Annual Meeting of the Canadian Society for Microscopy. Vancouver, BC, Canada. 22 May 2019)

Gault, B.; Breen, A. J.; Mouton, I.; Lu, W.; Wang, S.; Szczepaniak, A.; Kontis, P.; Stephenson, L.; Kwiatkowski da Silva, A.; Liebscher, C.; Herbig, M.; Raabe, D.; Britton, T. B.: *Atomic scale analysis of grain boundary deuteride growth front in Zircaloy-4* (TMS 2019. San Antonio, TX, USA. 10 Mar. 2019)

Gault, B.; Makineni, S. K.; He, J.; Lenz, M.; Neumeier, S.; Spiecker, E.; Raabe, D.: *Solute segregation effect at planar defects during creep of CoNi- & Co-based superalloys* (TMS 2019. San Antonio, TX, USA. 10 Mar. 2019)

Han, F.; Diehl, M.; Roters, F.; Raabe, D.: *Multi-scale modeling of plasticity* (ICIAM 2019 - The 9th International Congress on Industrial and Applied Mathematics. Valencia, Spain. 15 Jul. 2019)

Herbig, M.: *Joint Nanoscale Structural and Chemical Characterization by Correlative Atom Probe Tomography and Transmission Electron Microscopy* (Joint Workshop on Nano-Characterisation (4TU.HTM / M2i). Utrecht, The Netherlands. 28 Oct. 2019)

Hickel, T.: *Application of Density Functional Theory in the Context of Phase Diagram Modelling* (MSIT Winter School on Materials Chemistry. Castle Ringberg, Tegernsee. 03 Mar. 2019)

Hickel, T.; Dey, P.; McEniry, E.; Yao, M.; Herbig, M.; Lipińska-Chwałek, M.; Liebscher, C.; Mušić, D.; Hallstedt, B.; Haase, C.; Song, W.; Scheu, C.; Ponge, D.; Raabe, D.; Neugebauer, J.: κ carbide microstructures and the role of interfaces in high-Mn lightweight steels (High-Mn Steel 2019. Aachen, Germany. 31 Mar. 2019)

Hickel, T.; Dutta, B.; Stockem, I.; Körmann, F.; Neugebauer, J.: *Ab initio design strategies for NiMn-based FSMA* (6th Int. Conf. of Ferromagnetic Shape-Memory Alloys CFSMA. Prague, Czech Republic. 02 Jun. 2019)

Hickel, T.; Gupta, A.; Neugebauer, J.; Kavakbasi, B. T.; Buranova, Y.; Kulitskiy, V. A.; Wilde, G.; Divinski, S. V.: *Precipitate-induced nonlinearities of solute diffusion in Al-based alloys* (15th Diffusion in Solids and Liquids (DSL) Conference. Athens, Greece. 27 Jun. 2019)

Hickel, T.; Janßen, J.; Sözen, H. I.; Körmann, F.; Neugebauer, J.; Lysogorskiy, Y.; Drautz, R.: *Ab initio simulation of finite temperature phase stabilities: Concepts and application* (HetSys Launch Event. Warwick, UK. 09 Sep. 2019)

Hickel, T.; Janßen, J.; Sözen, H. I.; Körmann, F.; Surendralal, S.; Todorova, M.; Lysogorskiy, Y.; Drautz, R.; Neugebauer, J.: *High-throughput optimization of finite temperature phase stabilities: Concepts and application* (Spring Meeting of the German Physical Society (DPG). Regensburg, Germany. 4 Apr. 2019)

Hickel, T.; Zendegani, A.; Körmann, F.; Neugebauer, J.: *Energetics of non-stoichiometric stacking faults in Fe–Nb alloys: An ab initio study* (TMS 2019. San Antonio, TX, USA. 10 Mar. 2019)

Huber, L.; Hadian, R.; Grabowski, B.; Neugebauer, J.: *Computing and modelling solute-grain boundary interaction* (Canadian Materials Science Conference. Vancouver, Canada. 12 Jun. 2019)

Ikeda, Y.; Körmann, F.; Neugebauer, J.: *Impact of Interstitial Alloying of High Entropy Alloys from First Principles* (2019. San Antonio, TX, USA. 10 Mar. 2019)

Janßen, J.; Hickel, T.; Neugebauer, J.: *Automated ab-initio Determination of Materials Properties at finite Temperatures with pyiron* (CNLS Seminar. Los Alamos, NM, USA. 10 Dec. 2019)

Jeong, J.: *Advanced transmission electron microscopy of nanomaterials using In-situ TEM and precession electron diffraction* (Seminar, Korea Institute of Science and Technology (KIST). Seoul, South Korea. 04 Nov. 2019)

Jeong, J.: *Advanced transmission electron microscopy of nanomaterials using In-situ TEM and precession electron diffraction* (Seminar, Korea Institute of Materials Science (KIMS). Seoul, South Korea. 08 Nov. 2019)

Jeong, J.: *Advanced transmission electron microscopy of nanomaterials using In-situ TEM and precession electron diffraction* (Seminar, Korea Institute of Industrial Technology (KITECH). Seoul, South Korea. 13 Nov. 2019)

Jeong, J.; Kim, J.; Kiener, D.; Oh, S. H.: *In-situ TEM observation of twin-dominated deformation of Mg single crystals* (KSM Annual Fall Conference 2019. Gyeongju, South Korea. 14 Nov. 2019)

Kontis, P.; Lilensten, L.; Kürnsteiner, P.; Cervellon, A.; Cormier, J.; Raabe, D.; Gault, B.: *The effect of segregation of solutes at crystal defects on the mechanical performance of superalloys* (10th Pacific Rim International Conference on Advance Materials and Processing (PRICM-10). Xi'an, China. 18 Aug. 2019)

Kontis, P.; Makineni, S. K.; Wu, X.; Mianroodi, J. R.; Shanthraj, P.; Cormier, J.; Raabe, D.; Gault, B.: *Understanding deformation mechanisms in superalloys through atomic scale microanalysis* (TMS 2019. San Antonio, TX, USA. 10 Mar. 2019)

Körmann, F.; Ikeda, Y.; Srinivasan, P.; Neugebauer, J.; Grabowski, B.; Kostiuchenko, T.; Shapeev, A.: *Phase stability and mechanical properties of high entropy and chemically complex alloys* (MS&T Annual Meeting. Portland, OR, USA. 01 Oct. 2019)

Kürnsteiner, P.; Hariharan, A.; Jung, H. Y.; Peter, N. J.; Wilms, M. B.; Weisheit, A.; Barriobero-Vila, P.; Gault, B.; Raabe, D.; Jägle, E. A.: *Application of Atom Probe Tomography to Complex Microstructures of Laser Additively Manufactured Samples* (Microscopy & Microanalysis Conference. Portland, OR, USA. 04 Aug. 2019)

Li, Z.; Su, J.; Lu, W.; Luo, H.; Wang, Z.; Wu, X.; Raabe, D.: *Recent progresses in the understanding of metastable high-entropy alloys* (TMS 2019. San Antonio, TX, USA. 10 Mar. 2019)

Liebscher, C.: *Transmission electron microscopy - Characterization across the length scales* (Pre-conference workshop of the 26th International Symposium on Metastable, Amorphous and Nanostructured Materials 2019 (ISMANAM 2019). Chennai, India. 07 Jul. 2019)

Liebscher, C.; Meiners, T.; Harzer, T. P.; Freysoldt, C.; Dehm, G.: *Atomic scale phase separation tendencies in nanostructured copper alloys* (26th International Symposium on Metastable, Amorphous and Nanostructured Materials 2019 (ISMANAM 2019). Chennai, India. 08 Jul. 2019)

Liebscher, C.; Meiners, T.; Peter, N. J.; Frolov, T.; Dehm, G.: *Experimental discovery of grain boundary phase transformations unveiled by atomistic simulations* (PICS^3 2019 Meeting, Centre Interdisciplinaire de Nanoscience de Marseille. Marseille, France. 21 Jun. 2019)

Liebscher, C.; Meiners, T.; Peter, N. J.; Frolov, T.; Dehm, G.: *Experimental discovery of grain boundary phase transformations unveiled by atomistic simulations* (Colloquium of IIT Gandhinagar. Palaj, India. 05 Jul. 2019)

Liebscher, C.; Meiners, T.; Peter, N. J.; Frolov, T.; Dehm, G.: *Exploration of interfacial transitions by correlating atomic scale microscopy with atomistic simulations* (TMS 2019. San Antonio, TX, USA. 10 Mar. 2019)

Liebscher, C.; Stoffers, A.; Alam, M.; Lymperakis, L.; Cojocaru-Mirédin, O.; Gault, B.; Neugebauer, J.; Dehm, G.; Scheu, C.; Raabe, D.: *Asymmetric Line Segregation at Faceted Si Grain Boundaries* (TMS 2019. San Antonio, TX, USA. 10 Mar. 2019)

Liebscher, C.; Stoffers, A.; Alam, M.; Lymperakis, L.; Cojocaru-Mirédin, O.; Gault, B.; Neugebauer, J.; Scheu, C.; Raabe, D.; Meiners, T.; Frolov, T.; Dehm, G.: *How do grain boundaries transform on the atomic level?* (International Workshop on Advanced and In-situ Microscopies of Functional Nanomaterials and Devices, IAMNano 2019. Düsseldorf, Germany. 29 Oct. 2019)

Lim, J.; Kim, S.-H.; Sahu, R.; Aymerich Armengol, R.; Kasian, O.; Choi, P.-P.; Stephenson, L.; Gault, B.; Scheu, C.: *Detection of trace impurities and other defects in functional nanomaterials* (International Workshop on Advanced and In-situ Microscopies of Functional Nanomaterials and Devices, IAMNano 2019. Düsseldorf, Germany. 27 Oct. 2019)

Liu, C.; Garner, A.; Zhao, H.; Gault, B.; Prangnell, P.; Raabe, D.; Shanthraj, P.: *CALPHAD-informed phase-field modeling of grain boundary microchemistry and microstructure in Al–Zn–Mg–Cu alloys* (LightMAT 2019 - 3rd Conference & Exhibition on Light Materials. Manchester, UK. 05 Nov. 2019)

Lymperakis, L.: *Surface rehybridization and strain effects on the composition and the properties of ternary III Nitride alloys* (19th International Conference on Crystal Growth and Epitaxy. Keystone, CO, USA. 28 Jul. 2019)

Lymperakis, L.: *Surface rehybridization effects of B incorporation at GaN and AlN surfaces: A potential route to overcome bulk solubility limits* (4th International Workshop on Ultraviolet Materials and Devices. Saint Petersburg, Russia. 08 Sep. 2019)

Lymperakis, L.: *Ab-initio based investigations of surface reconstructions and strain effects on the compositional limits of ternary III-Nitride alloys* (National Institute of Advanced Industrial Science and Technology (AIST) Colloquium. Tsukuba, Japan. 09 Oct. 2019)

Lymperakis, L.: *Ab-initio based description and design of nitride surfaces: The role of surface rehybridization on alloy composition and ordering* (Materials Research Meeting 2019 (Materials Research Society of Japan). Yokohama, Japan. 10 Oct. 2019)

Mendive-Tapia, E.: *Ab-initio Free energy of antiferromagnetic phases: a hierarchy of local moment correlation functionsAb-initio Free energy of antiferromagnetic phases: a hierarchy of local moment correlation functions* (Current Research in Magnestism (CRIM) 2019: Recent advances in Antiferromagnetism. London, UK. 18 Sep. 2019)

Nandy, S.; Zaefferer, S.: *On the role of Ca, Zn and Al for ductilization of Mg alloys* (27th International Conference on Materials and Technology (27 ICM&T). Portoroz, Slovenia. 16 Oct. 2019)

Neugebauer, J.; Surendralal, S.; Todorova, M.: *Extending First-Principles Calculations to Model Electrochemical Reactions at the Solid-Liquid Interface* (Towards Reality in Nanoscale Materials X. Levi, Finnland. 12 Feb. 2019)

Neugebauer, J.; Janßen, J.; Körmann, F.; Hickel, T.; Grabowski, B.: *Exploiting large ab initio data spaces to design materials* (Opening of Christian Doppler Laboratory for nanoscale phase transformations. Linz, Austria. 28 Feb. 2019)

Neugebauer, J.; Todorova, M.; Grabowski, B.; Hickel, T.: *Modelling structural materials in realistic environments by ab initio thermodynamics* (Hume-Rothery Award Symposium, TMS 2019. San Antonio, TX, USA. 13 Mar. 2019)

Neugebauer, J.: *Automizing work flows in computational materials design* (Traceability and securing of results as essential challenges of research in the digital age. Berlin, Germany. 09 Apr. 2019)

Neugebauer, J.; Janßen, J.; Körmann, F.; Hickel, T.; Grabowski, B.: *Exploiting large ab initio data spaces to design materials with superior mechanical properties* (2019 BIOVIA User Conference. Düsseldorf, Germany. 15 May 2019)

Neugebauer, J.; Janßen, J.; Hickel, T.: *Automated uncertainty analysis and quantification for high-precision DFT calculations* (Workshop “Precision Quantification in DFT.” Louvaine-la-Neuve, Belgien. 23 May 2019)

Neugebauer, J.: *Machine Learning in Materials: Screening and Discovery* (Gordon Research Conference Physical Metallurgy „Coupling Computation, Data Science and Experiments in Physical Metallurgy“. Manchester, NH, USA. 08 Jul. 2019)

Neugebauer, J.; Huber, L.; Körmann, F.; Grabowski, B.; Hickel, T.: *Ab initio input for multiphysics models: Accuracy, performance and challenges* (ISAM4: The fourth International Symposium on Atomistic and Multiscale Modeling of Mechanics and Multiphysics. Erlangen, Germany. 05 Aug. 2019)

Neugebauer, J.: *Ab initio guided materials design and discovery* (Inaugural Symposium for Computational Materials, Skoltech. Moscow, Russia. 04 Sep. 2019)

Neugebauer, J.; Stockem, I.; Hegde, O.; Freysoldt, C.; Körmann, F.; Hickel, T.; Alling, B.: *Ab initio description of coupling phenomena between magnetic and structural degrees of freedom* (EASTMAG2019 – VII Euro-Asian Symposium "Trends in Magnetism." Jekaterinburg, Russia. 10 Sep. 2019)

Neugebauer, J.; Janßen, J.; Körmann, F.; Hickel, T.; Grabowski, B.: *Ab initio descriptors to design materials with superior mechanical properties* (Materials Day, ETH Zürich. Zürich, Switzerland. 20 Nov. 2019)

Neugebauer, J.: *Machine Learning in Materials: Screening and Discovery* (National Institute of Advanced Industrial Science and Technology (AIST). Tsukuba, Japan. 09 Dec. 2019)

Neugebauer, J.; Janßen, J.; Huber, L.; Körmann, F.; Hickel, T.; Grabowski, B.: *Construction and exploitation of large ab initio data spaces to design materials with superior mechanical properties* (MRS-J 2019. Yokohama, Japan. 11 Dec. 2019)

Neugebauer, J.; Janßen, J.; Körmann, F.; Hickel, T.; Grabowski, B.: *Exploration of large ab initio data spaces to design materials with superior mechanical properties* (Physics and Theoretical Division Colloquium. Los Alamos, NM, USA. 31 Jan. 2019)

Neugebauer, J.; Surendralal, S.; Todorova, M.: *First-principles approach to model electrochemical reactions at solid-liquid interfaces* (ACS 2019 Fall Meeting & Exhibition. San Diego, CA, USA. 25 Aug. 2019)

Peter, N. J.; Meiners, T.; Duarte, M. J.; Kirchlechner, C.; Frolov, T.; Liebscher, C.; Dehm, G.: *Grain boundary phase transformations in Cu: New insights by advanced STEM* (65. Metallkunde-Kolloquium. Lech am Arlberg, Austria. 23 Apr. 2019)

Raabe, D.: *Atomic-Scale Analysis of Chemistry at Lattice Defects* (The KAIST Lecture in Materials Science and Engineering 2019, Korea Advanced Institute of Science and Technology KAIST. Daejeon, Korea. 07 May 2019)

Raabe, D.: *Compositional Lattice Defect Manipulation for Microstructure Design* (The Bauerman Lecture 2019, Department of Materials, Imperial College London, Royal School of Mines. London, UK. 28 Feb. 2019)

Raabe, D.: *Metastable Nanostructured Metallic Alloy* (The KAIST Lecture in Materials Science and Engineering 2019, Korea Advanced Institute of Science and Technology KAIST. Daejeon, Korea. 07 May 2019)

Raabe, D.; Katnagallu, S.; Stephenson, L.; Balachandran, S.; Freysoldt, C.; Neugebauer, J.; Gault, B.: *Imaging single vacancies and atoms using joint FIM and APT experiments* (Conference on Possibilities and Limitations of Quantitative Materials Modeling and Characterization. Bernkastel-Kues, Germany. 20 May 2019)

Raabe, D.; Neugebauer, J.; Körmann, F.; Rao, Z.; Gault, B.; Lu, W.; Li, Z.: *Metastability High Entropy Alloy Design* (MRS Fall Meeting. Boston, MA, USA. 01 Dec. 2019)

Raabe, D.; Ponge, D.; Kwiatkowski da Silva, A.; Katnagallu, S.; Stephenson, L.; Freysoldt, C.; Diehl, M.; Liebscher, C.; Kamachali, R. D.; Zaefferer, S.; Neugebauer, J.; Sandlöbes, S.; Gault, B.; Scheu, C.; Roters, F.: *From Atomistic Understanding to Engineering Design of Advanced Medium and High Manganese Steels* (4th International Conference on medium and high Manganese steels. Aachen, Germany. 01 Apr. 2019)

Raabe, D.; Ponge, D.; Kwiatkowski da Silva, A.; Makineni, S. K.; Katnagallu, S.; Stephenson, L.; Freysoldt, C.; Neugebauer, J.; Gault, B.: *Chemistry and Structure of Lattice Defects* (Physics Colloquium, Faculty of Physics, University Duisburg-Essen. Duisburg, Germany. 15 May 2019)

Raabe, D.; Ponge, D.; Kwiatkowski da Silva, A.; Makineni, S. K.; Katnagallu, S.; Stephenson, L.; Kontis, P.; Freysoldt, C.; Neugebauer, J.; Gault, B.: *Chemistry at Lattice Defects Probed at Atomic Scale* (20th International Union of Materials Research Societies International Conference in Asia IUMRS. Perth, Australia. 22 Sep. 2019)

Raabe, D.; Ponge, D.; Kwiatkowski da Silva, A.; Makineni, S. K.; Katnagallu, S.; Stephenson, L.; Kontis, P.; Wu, X.; Freysoldt, C.; Neugebauer, J.; Gault, B.: *Chemistry at Lattice Defects Probed at Atomic Scale* (The 53rd Annual Meeting of the Israel Society for Microscopy, Tel Aviv, Israel. Tel Aviv, Israel. 29 May 2019)

Raabe, D.; Ponge, D.; Kwiatkowski da Silva, A.; Makineni, S. K.; Katnagallu, S.; Stephenson, L.; Kontis, P.; Wu, X.; Freysoldt, C.; Neugebauer, J.; Gault, B.: *Interaction of Chemistry and Structure at Lattice Defects at Atomic Scale* (Russell Berrie Nanotechnology Institute, Technion, Haifa, Israel. Haifa, Israel. 28 May 2019)

Raabe, D.; Ponge, D.; Li, Z.; Neugebauer, J.; Körmann, F.; Rao, Z.; Gault, B.: *Metastable High Entropy Alloys* (World Congress on High Entropy Alloys. Seattle, WA, USA. 18 Sep. 2019)

Rohwerder, M.: *Die Kelvinsondentechnik in der Korrosion: von der Grundlagenforschung bis hin zu potentiellen Anwendungen im Feld* (ProcessNet Meeting "Elektrochemische Prozesse", Dechema-Haus. Frankfurt, Germany. 21 Jan. 2019)

Rohwerder, M.: *Hydrogen embrittlement, hydrogen traps, high sensitive detection of hydrogen with high spatial resolution, corrosion* (Dreiländerkorrosionstagung, Dechema-Haus. Frankfurt, Germany. 09 Apr. 2019)

Rohwerder, M.: *Intelligent coatings for corrosion protection: on the need for new coating concepts* (International Conference on Corrosion Protection and Application (ICCPA 2019). Chongqing, China. 10 Oct. 2019)

Rohwerder, M.: *Scanning Kelvin Probe based techniques for mapping hydrogen distribution in metals and their application for investigating hydrogen embrittlement* (Workshop "Hydrogen in Metals", St Anne's College. Oxford, UK. 15 Apr. 2019)

Rohwerder, M.: *Zinc alloy coatings and nano-composite coatings for corrosion protection: From the basics to new challenges* (IIM NMD ATM 2019: Advanced Materials for Industrial and Societal Applications. Kovalam, Thiruvananthapuram, India. 13 Nov. 2019)

Roters, F.: *DAMASK - The Düsseldorf Advanced MAterial Simulation Kit for studying multi-physics crystal plasticity phenomena* (TOOLKIT Seminar Integrated Computational Materials Engineering, OCAS. Gent, Belgium. 09 Jul. 2019)

Roters, F.; Han, F.: *Calibrating yield surface models based on full yield crystal plasticity simulations* (M2i conference "Meeting Materials." Noordwijkerhout, The Netherlands. 10 Dec. 2019)

Scheu, C.: *Advanced Cs corrected STEM imaging coupled to 3D atom probe tomography* (SCANDEM 2019. Gothenburg, Sweden. 12 Jun. 2019)

Scheu, C.: *Materials for renewable energy applications* (Metallurgical Engineering and Materials Science Department Colloquium, Indian Institute of Technology. Mumbai, India. 15 Jul. 2019)

Scheu, C.; Folger, A.: *Annealing treatment in various atmospheres: A tool to control structure and properties of* TiO_2 *nanowires* (6th International Symposium on Metastable, Amorphous and Nanostructured Materials (ISMANAM-2019). Chennai, India. 08 Jul. 2019)

Scheu, C.; Hieke, S. W.: *Fundamentals and Applications of Electron Energy-Loss Spectroscopy in a Scanning Transmission Electron Microscope* (Universita' Roma Tre Colloquium. Roma, Italy. 25 Jul. 2019)

Scheu, C.; Hieke, S. W.: *How stable are thin Aluminium films: Dewetting phenomena observed by in-situ electron microscopy* (Microscopy Conference 2019 (MC2019). Berlin, Germany. 01 Sep. 2019)

Scheu, C.; Zhang, S.: *Effect of interfaces on the photoelectrochemical performance of functional oxides* (PICS^3 2019 Meeting, Centre Interdisciplinaire de Nanoscience de Marseille. Marseille, France. 21 Jun. 2019)

Shah, V.; Diehl, M.; Roters, F.: *Prediction of Nucleation Sites for Recrystallization using Crystal Plasticity Simulations* (7th International Conference on Recrystallization and Grain Growth. Ghent, Belgium. 04 Aug. 2019)

Stein, F.: *Experimental Determination of Phase Diagrams* (3rd MSIT Winter School on Materials Chemistry. Castle Ringberg, Tegernsee. 04 Mar. 2019)

Stein, F.: *Fe–Al-based Materials: Phase Diagrams, Properties, and Potential for Applications* (Seminar, Hokkaido University of Science. Sapporo, Japan. 28 Oct. 2019)

Stein, F.: *Stability Competition between Laves Phase Polytypes* (Tokyo Institute of Technology. Tokyo, Japan. 01 Nov. 2019)

Stein, F.: *The Co–Ti system revisited: About the cubic-to-hexagonal Laves phase transformation and other controversial features of the phase diagram* (Seminar, Hokkaido University. Sapporo, Japan. 29 Oct. 2019)

Stein, F.; Luo, W.; Kirchlechner, C.; Dehm, G.: *Micromechanics of Laves Phases: Strength, Fracture Toughness, and Hardness as Function of Composition and Crystal Structure* (Joint EPRI-123 HiMAT Conference on Advances in High Temperature Materials. Nagasaki, Japan. 20 Oct. 2019)

Stephenson, L.; Katnagallu, S.; Mouton, I.; Oliveira, F.; Gault, B.; Raabe, D.: *An Atomic Renaissance for Pulsed Field Ion Microscopy* (Microscopy & Microanalysis 2019. Portland, OR, USA. 04 Aug. 2019)

Sun, B.; Krieger, W.; Ponge, D.; Rohwerder, M.; Raabe, D.: *Hydrogen embrittlement of medium Mn steels with ferrite matrix or austenite matrix* (4th High Manganese Steel 2019 HMnS. Aachen, Germany. 01 Apr. 2019)

Surendralal, S.: *Implementation of an ab initio electrochemical potentiostat: Application to Mg corrosion* (Symposium "Fundamentals of the electrochemistry of the metal/electrolyte interface", Imperial College. London, UK. 25 Apr. 2019)

Tehranchi, A.; Hickel, T.; Neugebauer, J.: *Atomistic simulations of hydrogen-defect interactions in metals* (Workshop "Hydrogen in Metals - current understanding and future needs", St Anne's College. Oxford, UK. 18 Apr. 2019)

Todorova, M.; Yoo, S.-H.; Surendralal, S.; Neugebauer, J.: *Modelling electrochemical solid/liquid interfaces by first principles calculations* (19th International Workshop on Computational Physics and Material Science: Total Energy and Force Methods, ICTP. Trieste, Italy. 10 Jan. 2019)

Todorova, M.; Yoo, S.-H.; Surendralal, S.; Neugebauer, J.: *A fully ab initio approach to electrochemistry and corrosion* (CNLS Colloquium, Los Alamos National Laboratory. Los Alamos, NM, USA. 28 Jan. 2019)

Todorova, M.; Surendralal, S.; Neugebauer, J.: *First-principles approach to model electrochemical reactions at the solid-liquid interface* (Spring Meeting of the German Physical Society (DPG). Regensburg, Germany. 03 Apr. 2019)

Todorova, M.; Yoo, S.-H.; Surendralal, S.; Neugebauer, J.: *A fully ab initio approach to modelling electrochemical solid/liquid interfaces* (Chemiekolloquium der Johannes Kepler Universität Linz. Linz, Austria. 14 May 2019)

Todorova, M.; Surendralal, S.; Neugebauer, J.: *Building an ab-initio potentiostat in a standard DFT code with periodic boundary conditions* (ELRC2019 - IPAM reuniuon workshop. Lake Arrowhead, CA, USA. 14 Jun. 2019)

Todorova, M.; Yoo, S.-H.; Surendralal, S.; Neugebauer, J.: *Ab initio approach to electrochemisty and corrosion* (Computational Materials Chemistry Workshop. Telluride, CO, USA. 16 Jul. 2019)

Todorova, M.; Surendralal, S.; Neugebauer, J.: *Degradation processes at surfaces and interfaces* (ISAM4: The fourth International Symposium on Atomistic and Multiscale Modeling of Mechanics and Multiphysics, Friedrich-Alexander Universität Erlangen-Nürnberg (FAU). Erlangen, Germany. 07 Aug. 2019)

Todorova, M.; Yoo, S.-H.; Surendralal, S.; Neugebauer, J.: *Predicting atomic structure and chemical reactions at solid-liquid interfaces by first principles* (Operando surface science – Atomistic insights into electrified solid/liquid interfaces (708. WE-Heraeus-Seminar), Physikzentrum. Bad Honnef, Germany. 10 Dec. 2019)

Zaefferer, S.: *Electron diffraction techniques in scanning electron microscopy: fundamentals and state-of-the-art applications of electron backscatter diffraction (EBSD) and electron channelling contrast imaging (ECCI)* (27th Annual Meeting of the German Crystallographic Society (DGK). Leipzig, Germany. 25 Mar. 2019)

Zaefferer, S.: *In-situ electron channeling contrast imag-ing (ECCI) to observe the effect of hydro-gen in TWIP steels and superalloys* (Physikalisches Kolloquium der Universität Wien. Wien, Austria. 29 Jan. 2019)

Zaefferer, S.: *Investigation on the effect of hydrogen on dislocation patterns in high-strength steels using electron channeling contrast imaging in the scanning electron microscope* (15th Multinational Congress on Microscopy. Belgrade, Serbia. 15 Sep. 2019)

Zaefferer, S.: *Measurement of local residual stresses using cross-correlation EBSD and ring core milling* (27th International Conference on Materials and Technology (26 ICM&T). Portoroz, Slovenia. 16 Oct. 2019)

Zaefferer, S.: *Microstructure Characterization in 2D and 3D using Advanced SEM-based Electron Diffraction Techniques* (3rd Materials Genome Engineering Forum. Kunming, China. 23 Nov. 2019)

Zaefferer, S.: *Microstructure Characterization in 2D and 3D using Advanced SEM-based Electron Diffraction Techniques* (Chongqing University Colloquium. Chongqing, China. 26 Nov. 2019)

Zaefferer, S.: *Residual stress measurements on steel using cross-correlation EBSD and ring core milling* (EDAX workshop. Weiterstadt, Germany. 30 Oct. 2019)

Zaefferer, S.: *Understanding hydrogen-embrittlement during fatigue loading of a high-Mn-steel using ECCI and CC-EBSD* (RMS-EBSD conference. London, UK. 03 Apr. 2019)

Zaefferer, S.; An, D.: *Hydrogen-induced embrittlement during fatigue loading of a high-Mn steel investigated by electron channelling contrast imaging (ECCI)* (Euromat 2019. Stockholm, Sweden. 01 Sep. 2019)

Zaefferer, S.; Nandy, S.; Sandlöbes, S.; Raabe, D.: *Understanding the ductilitization effect in Mg alloyed with Ca, Al, and Zn* (Euromat 2019. Stockholm, Sweden. 01 Sep. 2019)

Zaefferer, S.; Shan, Y.; Madivala, M.: *Nano-indentation and electron channeling contrast imaging (ECCI) to understand the interaction of hydrogen and dislocations in a high-Mn TWIP steel* (Euromat 2019. Stockholm, Sweden. 01 Sep. 2019)

Zhang, S.: *Electron Microscopy* (DGK-AK20 Summer School “Synthesis and characterization of inorganic functional materials.” Mülheim (Ruhr), Germany. 22 Jul. 2019)

Zhang, S.: *Modern electron microscopy goes high dimensions: handling big data* (BIG DATA SUMMER – BiGmax Network. Platja d’Aro, Spain. 09 Sep. 2019)

2020

Antonov, S.: *Overview of the Damage Accumulation Mechanisms During Non-isothermal Creep of Ni-based superalloys* (Seminar, Exponent, online. Atlanta, GA, USA. 18 Dec. 2020)

Antonov, S.; Tan, Q.; Gault, B.: *Atom Probe Tomographic Investigation of the Solute Segregation to Crystal Defects in γ-phase Co–35Ni–20Cr–10Mo Superalloy* (Microscopy & Microanalysis (M&M) Meeting, virtual. Milwaukee, WI, USA. 01 Aug. 2020)

Best, J. P.: *Connecting structure to the micro-mechanics and macro-scale fracture toughness of a laser-processed BMG using micro-focussed high-energy X-rays at PETRA III* (PETRA IV Workshop – Earth, Environment, and Materials for Nanoscience and Information Technology, online. Hamburg, Germany. 02 Nov. 2020)

Best, J. P.: *Nano-/Micromechanics of Materials: A focus on laser-processed BMGs* (Deutsches Zentrum für Luft- und Raumfahrt (DLR) Seminar Series, online. Köln, Germany. 01 Dec. 2020)

Best, J. P.: *Small-scale mechanics at the Max-Planck-Institute in Düsseldorf: An overview* (Oxford Materials Group Seminar Series, online. Oxford, UK. 27 Aug. 2020)

Dehm, G.: *Micro- and Nanomechanical Testing of Materials - From Methods to Mechanical Properties* (SFB 1394 Summer School, online. Aachen, Germany. 05 Oct. 2020)

Dehm, G.: *Resolving grain boundary phase transformations in Copper by advanced STEM* (TimeMan, online. Lille, France. 22 Oct. 2020)

Diehl, M.; Cereceda, D.; Marian, J.; Liu, C.; Dong, J.; Wicke, M.; Brückner-Foit, A.; Kamachali, R. D.; Shanthraj, P.; Roters, F.; Eisenlohr, P.; Raabe, D.: *Coupling Crystal Plasticity and Phase Field Methods - The Future of Integrated Computational Materials Engineering?* (Seminar “Materials Technology, in particular of Magnesium Materials.” Geesthacht, Germany. 17 Jan. 2020)

Diehl, M.; Kusampudi, N.; Kusche, C.; Raabe, D.; Korte-Kerzel, S.: *Combining Experiments, Simulations, and Data Science to Understand Damage in Dual Phase Steels* (International Conference on Plasticity, Damage, and Fracture. Riviera May, Mexico. 03 Jan. 2020)

Duarte, M. J.; Fang, X.; Rao, J.; Dehm, G.: *Hydrogen-microstructure interactions at small scale by in-situ nanoindentation during hydrogen charging* (Nanobrücken 2020: A nanomechanical Testing Conference. Düsseldorf, Germany. 05 Feb. 2020)

Eisenlohr, P.; Chakraborty, A.; Shanthraj, P.; Diehl, M.; Pagan, D.; Bieler, T. R.: *More than Crystal Plasticity: Multi-*

physics in DAMASK (TMS 2020. San Diego, CA, USA. 23 Jan. 2020)

Gault, B.: *An introduction to atom probe tomography: from fundamentals to atomic-scale insights into engineering materials* (Rolls Royce Lunch Time Seminar. Derby, UK. 01 Jul. 2020)

Gault, B.: *Pushing the limits of atomic-scale analytical microscopy* (London Centre for Nanoscience Lunch Time Seminar, online. London, UK. 11 Nov. 2020)

Gault, B.; Kim, S.-H.; Lim, J.; El-Zoka, A.; Kasian, O.; Sahu, R.; Stephenson, L.; Scheu, C.: *Nanoparticle Specimen Preparation for Atom Probe: Chemical Fixation and… cryo-Fixation (?)* (TMS 2020. San Diego, CA, USA. 23 Feb. 2020)

Gault, B.; Kwiatkowski da Silva, A.; Zhao, H.; Ponge, D.; Raabe, D.: *Atom probe tomography for studying the interplay of segregation and phase transformations* (17th International Conference on Aluminium Alloys, online. Grenoble, France. 26 Oct. 2020)

Güder, Ü.: *Archaeo-metallurgical Studies on Objects and Slags from the Persian period Iron Smithy in Tel Akko* (Early Iron Production: Experimental Archaeology, The Southern Levant and Africa, Research Workshop of the Israel Science Foundation. Ariel, Israel. 02 Feb. 2020)

Herbig, M.: *Material alterations in intense mechanical and chemical contacts* (TMS 2020. San Diego, CA, USA. 23 Feb. 2020)

Hickel, T.; Aydin, U.; Sözen, H. I.; Dutta, B.; Pei, Z.; Neugebauer, J.: *Innovative concepts in materials design to boost renewable energies* (Seminar of Institute for Innovative Technologies, SRH Berlin University of Applied Sciences. Berlin, Germany. 15 Jan. 2020)

Hickel, T.; McEniry, E.; Nazarov, R.; Dey, P.: *Ab initio basierte Simulation zur Wasserstoffversprödung in hoch-Mn Stählen* (Seminar der Staatlichen Materialprüfungsanstalt Darmstadt, Institut für Werkstoffkunde. Darmstadt, Germany. 21 Jan. 2020)

Hickel, T.: *Application of Density Functional Theory in the Context of Phase Diagram Modelling* (MSIT Winter School on Materials Chemistry, Virtual Event. Castle Ringberg, Tegernsee. 16 Feb. 2020)

Hickel, T.; Janßen, J.; Sözen, H. I.; Körmann, F.; Neugebauer, J.: *High-throughput optimization of finite temperature phase stabilities: Concepts and application* (IMWF Stuttgart, Colloquium Materials, virtual. Stuttgart, Germany. 10 Dec. 2020)

Janßen, J.; Hickel, T.; Neugebauer, J.: *Automated ab-initio Determination of Materials Properties at finite Temperatures with pyiron* (NIST Workshop, virtual. Rockville, MD, USA. 06 Aug. 2020)

Janßen, J.; Hickel, T.; Neugebauer, J.: *pyiron – an integrated development environment for ab initio thermodynamics* (AMS Seminar, virtual. Bochum, Germany. 05 Oct. 2020)

Körmann, F.; Ikeda, Y.; Srinivasan, P.; Dutta, B.; Neugebauer, J.; Grabowski, B.; Kostiuchenko, T.; Shapeev, A.: *Ab Initio Phase Stabilities of High Entropy and Chemically Complex Alloys* (TMS 2020. San Diego, CA, USA. 27 Feb. 2020)

Liebscher, C.: *Strain and temperature induced phase transformations in high entropy alloys explored by in situ S/TEM* (Institute of Micro- and Nanostructure Research Colloquium, Friedrich-Alexander University Erlangen-Nürnberg, online. Erlangen, Germany. 25 Jun. 2020)

Liebscher, C.; Lu, W.; Dehm, G.; Raabe, D.; Li, Z.: *Complex phase transformation pathways in high entropy alloys explored by in situ S/TEM* (Third International Conference on High Entropy Materials. Berlin, Germany. 26 Sep. 2020)

Liebscher, C.; Lu, W.; Li, Z.; Raabe, D.; Dehm, G.: *Atomic scale in situ observations of phase transformations in complex alloy systems* (Integrated in-situ solutions for TEM – DENSsolutions, TVIPS and QuantumDesign Workshop. Gauting, Germany. 12 Feb. 2020)

Liebscher, C.; Meiners, T.; Peter, N. J.; Frolov, T.; Dehm, G.: *Atomic resolution observations of grain boundary phase transformations* (Gaseous Electronics Symposium (GES3). Rogla, Slovenia. 05 Feb. 2020)

Lymperakis, L.: Nanomaterials from first principles (Department of Physics Colloquium, University of Crete, delivered online. Crete, Greece. 05 Nov. 2020)

Lymperakis, L.: *Properties and design of nanomaterials from first principles* (Department of Physics Colloquium, University of Crete, delivered online. Crete, Greece. 05 Nov. 2020)

Mendive-Tapia, E.: *Ab initio origin of the short period magnetism of MnGe from thermally fluctuating local moments* (Quantum Theory of Materials Seminar, virtual. Jülich, Germany. 08 Jul. 2020)

Neugebauer, J.; Janßen, J.; Huber, L.; Ikeda, Y.; Körmann, F.; Grabowski, B.; Hickel, T.; Shapeev, A.: *Materials Design in High Dimensional Chemical and Structural Configuration Spaces* (TMS 2020. San Diego, CA, USA. 27 Feb. 2020)

Neugebauer, J.; Lymperakis, L.; Janßen, J.; Huber, L.; Hickel, T.: *Modeling crystal growth and materials design in high dimensional chemical and structural configuration spaces* (German Conference on Crystal Growth DKT 2020. München/Garching, Germany. 12 Mar. 2020)

Palm, M.: *Phase Equilibria and Phase Diagrams* (4th MSIT Winter School on Materials Chemistry. Online. 17 Feb. 2020)

Palm, M.: *Eisenaluminide* (DGM-WEBinar „Einführung in metallische Hochtemperaturwerkstoffe. Online. 13 May 2020)

Ponge, D.; Kwiatkowski da Silva, A.; Makineni, S. K.; Katnagallu, S.; Zhou, X.; Mianroodi, J. R.; Stephenson, L.; Kontis, P.; Freysoldt, C.; Gault, B.; Neugebauer, J.: *Chemistry and Structure at Lattice Defects studied at the Atomic Scale* (4th Symposium Core Research Cluster Materials

Science & 3rd Symposium on International Joint Graduate Program in Materials Science, Tohoku University, online. Tohoku, Japan. 16 Nov. 2020)

Raabe, D.: *Interplay of Chemistry and Structure at Lattice Defects studied at the Atomic Scale* (The 4th Symposium for The Core Research Cluster for Materials Science and the 3rd Symposium on International Joint Graduate Program in Materials Science "Create New Value of Materials Science through Broad Collaboration", Tohoku University, online. Sendai, Japan. 16 Nov. 2020)

Raabe, D.: *Theory-guided design of materials, microstructures and processes* (Workshop on the Future of Materials Science, Institute of Nanotechnology, KIT, online. Karlsruhe, Germany. 08 Jul. 2020)

Raabe, D.; Diehl, M.; Shanthraj, P.; Sedighiani, K.; Roters, F.: *Multi-scale and multi-physics simulations of chemo-mechanical crystal plasticity problems for complex engineering materials using DAMASK* (Online Colloquium Lecture, Department of Materials Science and Engineering, KTH Royal Institute of Technology. Stockholm, Sweden. 05 Jun. 2020)

Raabe, D.; Kim, S.-H.; Zhang, X.; Schweinar, K.; Souza Filho, I. R.; Ma, Y.; Stephenson, L.; El-Zoka, A.; Mianroodi, J. R.; Rohwerder, M.; Lilensten, L.; Paolantonio, M.; Thoudden Sukumar, P.; Schneider, J. M.; Ponge, D.; Gault, B.: *Basic Research Opportunity and Leverage Effects in Sustainable Metallurgy* (Workshop: New Frontiers in Materials Science and Engineering – Sustainable Metallurgical Processes with improved CO_2 efficiency, RWTH Aachen, online. Aachen, Germany. 15 Sep. 2020)

Raabe, D.; Li, Z.; Ikeda, Y.; Dutta, B.; Körmann, F.; Neugebauer, J.: *Thermodynamics and symmetry effects in high entropy alloys* (Third International Conference on High Entropy Materials. Berlin, Germany. 26 Sep. 2020)

Ramachandramoorthy, R.: *High strain rate micromechanics: Instrumentation and implementation* (DGM - Arbeitskreis Rasterkraftmikroskopie und nanomechanische Methoden, online. 08 Jul. 2020)

Rohwerder, M.: *Strategies for enhancing Corrosion Protection by Organic Coatings* (71st Annual Meeting of the International Society of Electrochemistry, virtual. 31 Aug. 2020)

Roters, F.; Diehl, M.; Eisenlohr, P.; Shanthraj, P.: *DAMASK: the Düsseldorf Advanced MAterial Simulation Kit for studying multi-field crystal plasticity phenomena* (Seminar, Yanshan University, online. Qinhuangdao, Hebei, China. 09 Dec. 2020)

Roters, F.; Diehl, M.; Sedighiani, K.: *(Re-) formulation of dislocation density based crystal plasticity models in view of insights from parameter determination* (Oberwolfach Workshop: Mechanics of Materials: Towards Predictive Methods for Kinetics in Plasticity, Fracture, and Damage. Oberwolfach, Germany. 10 Mar. 2020)

Sahu, R.: *Study of phase impurity, 2D and 3D defects in orthorhombic MoAlB MAB phase* (Advances in Correlative Microscopy Workshop, IIT Madras. Chennai, India. 31 Jan. 2020)

Scheu, C.: *Atomic-scale characterization of complex solid solution nanoparticles using TEM* (Workshop on High Entropy Alloy and Complex Solid Solution Nanoparticles for Electrocatalysis, RUB, online. Bochum, Germany. 06 Oct. 2020)

Sedighiani, K.; Traka, K.; Diehl, M.; Roters, F.; Bos, K.; Sietsma, J.; Raabe, D.: *A Coupled Crystal Plasticity – Cellular Automaton Method for 3D Modeling of Recrystallization: Part I: Crystal Plasticity* (International Conference on Plasticity, Damage, and Fracture. Riviera May, Mexico. 03 Jan. 2020)

Stein, F.: *Experimental Determination of Phase Diagrams* (Lecture, 4th MSIT Winter School on Materials Chemistry. Castle Ringberg, Tegernsee. 16 Feb. 2020)

Stein, F.; Distl, B.; Palm, M.; Hauschildt, J.; Rackel, M. W.; Pyczak, F.; Mayer, S.; Yang, Y.; Chen, H.-L.; Engström, A.: *Improvement of a CALPHAD Database for the Development of Next Generation TiAl Alloys by Targeted Key Experiments on High-temperature Phase Equilibria – The EU Project ADVANCE* (Hume-Rothery Symposium: Phase Equilibria and Kinetics for Materials Design and Engineering, TMS 2020. San Diego, CA, USA. 23 Feb. 2020)

Todorova, M.: *Panel discussion leader* (BMBF-DOE bilateral workshop *Artificial Photosynthesis*, Virtual Discussion. Berlin, Germany. 24 Jun. 2020)

Todorova, M.; Yoo, S.-H.; Surendralal, S.; Neugebauer, J.: *Insights into the stability and reactivity of solid/liquid interfaces from ab initio calculations* (71st Annual Meeting of the International Society of Electrochemistry "Electrochemistry towards Excellence", virtual. Belgrade, Serbia. 03 Sep. 2020)

Traka, K.; Sedighiani, K.; Bos, K.; Lopez, J. G.; Diehl, M.; Angenendt, K.; Kestens, L.; Raabe, D.; Sietsma, J.: *A Coupled Crystal Plasticity – Cellular Automaton Method for 3D Modeling of Recrystallization: Part II: Subgrain Growth for Recrystallization* (International Conference on Plasticity, Damage, and Fracture. Riviera May, Mexico. 03 Jan. 2020)

Vercik, M.; Güder, Ü.: *Korrodiert, fragmentiert, zerbrochen. Die Aussagekraft der Eisenfunde aus dem Apollon-Heiligtum von Didyma* (Universität Hamburg, Archäologie und Kulturgeschichte des Antiken Mittelmeerraumes. Hamburg, Germany. 02 Dec. 2020)

Yoo, S.-H.: *Investigations of materials surfaces based on density functional theory calculations* (Department of Chemistry Seminar, Kangwon University, virtual. Chuncheon, Republic of Korea. 19 Nov. 2020)

Zaefferer, S.: *Combination of 2D and 3D SEM-based diffraction techniques with various other techniques for understanding of microstructures* (Workshop on correlative microscopy. Chennai, India. 31 Jan. 2020)

Zaefferer, S.: *Electron Channelling Contrast Imaging (ECCI) – A Technique for Observation and Quantitative In-situ Characterization of Crystal Lattice Defects in Bulk Samples* (12th Asia-Pacific Microscopy Conference (APMC 2020). Hyderabad, India. 03 Feb. 2020)

Zaefferer, S.: *Investigations on microstructural reasons for Goss texture formation in GO electrical steels - a search for the needle in the haystack* (9th International Conference Magnetism and Metallurgy. Rome, Italy. 03 Nov. 2020)

Zaefferer, S.: *Microstructure Characterization in 2D and 3D using Advanced SEM-based Electron Diffraction Techniques* (IISC Colloquium. Bangalore, India. 07 Feb. 2020)

Zaefferer, S.: *Towards understanding hydrogen embrittlement: Tools for microscopic and nanoscopic detection of hydrogen and its mechanical effects in microstructures of steels and superalloys* (International e-Conference on Structural Materials for Nuclear and Space Applications (SNSA20). Mumbai, India. 03 Dec. 2020)

Zhang, S.: *Efficiency and stability of catalytic nanostructures* (Colloquium, Ludwig-Maximilians-Universität. München, Germany. 13 Nov. 2020)

Zhao, H.; Gault, B.; De Geuser, F.; Huber, L.; Lu, W.; Peter, N. J.; Ponge, D.; Raabe, D.: *Segregation and precipitation at grain boundaries in an Al-Zn-Mg-Cu alloy* (17th International Conference on Aluminium Alloys ICAA 2020, virtual. Grenoble, France. 26 Oct. 2020)

Zhou, X.; Mianroodi, J. R.; Kwiatkowski da Silva, A.; Koenig, T.; Thompson, G. B.; Shanthraj, P.; Ponge, D.; Gault, B.; Svendsen, B.; Raabe, D.: *Revealing the dependence of Au segregation on dislocation character in Pt–Au via cross-correlative microscopy* (APT&M 2020, virtual. Oxford, UK. 16 Nov. 2020)

2021

Antonov, S.: *A primer to atom probe tomography and its application to intermetallics* (Intermetallics 2021. Bad Staffelstein, Germany. 04 Oct. 2021)

Antonov, S.: *Towards Improved Superalloy Performance via Defect Engineering* (Department of Mechanical Colloquium, Industrial, and Manufacturing Engineering, Oregon State University, online. Corvallis, OR, USA. 10 May 2021)

Antonov, S.: *Understanding phase transformations at boundaries and interfaces in β-Titanium alloys at the near-atomic scale* (Conference on Possibilities and Limitations of Quantitative Materials Modeling and Characterization. Bernkastel-Kues, Germany. 12 Oct. 2021)

Antonov, S.: *Understanding Superalloys on the Atomic Scale* (Department of Materials Science Colloquium, University of Illinois Urbana-Champaign, online. Urbana, IL, USA. 01 Mar. 2021)

Antonov, S.: *Understanding the Defect-Solute Interactions during Deformation of Superalloys* (Colloquium, Oak Ridge National Laboratory, online. Oak Ridge, TN, USA. 25 Jun. 2021)

Antonov, S.; Shi, R.; Li, D.; Kloenne, Z.; Zheng, Y.; Fraser, H. L.; Raabe, D.; Gault, B.: *Atom Probe Tomographic Study of Precursor Metastable Phases and Their Influence on a Precipitation in the Metastable ß-titanium Alloy, Ti–5Al–5Mo–5V–3Cr* (TMS 2021, online. Pittsburgh, PA, USA. 14 Mar. 2021)

Brink, T.: *From contact mechanics to wear* (EPFL Summer School: Tribology and Surfaces Interactions. Visp, VS, Switzerland. 23 Aug. 2021)

Dehm, G.: *Congruent and non-congruent grain boundary phase transformations in Copper studied by advanced STEM* (Virtual Seminar of Institute Jozef Stefan. Ljubljana, Slovenia. 04 Mar. 2021)

Dehm, G.: *Experimental Insights in Congruent and Non-Congruent Grain Boundary Phase Transformations in Copper by Advanced STEM* (International Seminars, Technion - Israel Institute of Technology (Israel), Purdue University (USA), virtual. 06 May 2021)

Dehm, G.: *Probing the local influence of micro- and nanostructure on mechanical properties of Materials* (E-MRS Spring Meeting, online. 31 May 2021)

G. Dehm: *Probing The Local Influence Of Materials' Nanostructure On Mechanical Properties* (Anhui University of Technology, School of Metallurgical Engineering, Ma'anshan, China, online. 04 Nov. 21)

G. Dehm, T. Meiners, J. Duarte, T. Frolov, C. Liebscher: *Atomic structure of two phases of a Cu tilt grain boundary resolved by scanning transmission electron microscopy* (MS&T, Columbus, USA, hybrid. 17 Oct. 21)

Dehm, G.; Peter, N. J.; Meiners, T.; Frommeyer, L.; Brink, T.; Frolov, T.; Liebscher, C.: *Grain boundary phases in pure and alloyed Cu: Insights from advanced STEM* (Microscopy Conference 2021 Joint Meeting of Dreiländertagung & Multinational Congress on Microscopy, Virtual Meeting. 22 Aug. 2021)

Diehl, M.; Kusampudi, N.: *Using machine learning and crystal plasticity simulation to design damage resistant dual phase steels* (Webinar: Metal Plasticity Seminar-Artificial Intelligence, Machine Learning and Big Data in Metal Plasticity. Leuven, Belgium. 01 Oct. 2021)

Duarte, M. J.: *Hydrogen effects on the mechanical behavior of Fe alloys at small scale* (Micromechanical and Macroscopic Modelling (MMM) Special Seminar, Interdisciplinary Centre for Advanced Materials Simulation (ICAMS), Ruhr-Universität Bochum, online. Bochum, Germany. 13 Jul. 2021)

Duarte, M. J.: *Metales y aleaciones bajo impacto ambiental: corrosión, fragilización por hidrógeno y propiedades mecánicas* (Primer Simposio "Materiales aplicados" del Capítulo Estudiantil de la Sociedad Mexicana de Materiales, Unidad Profesional Interdisciplinaria de Ingeniería, Campus Guanajuato, UPIIG IPN, online. Guanajuato, Mexico. 25 May 2021)

Duarte, M.J.; Rao, J.; Dehm, G.: *Hydrogen and micromechanics in ferritic alloys* (Oxford Materials Colloquium, online seminar. Oxford, United Kingdom. 4 Feb. 2021)

El-Zoka, A.: *Materials Degradation: Turning a problem into a solution* (Colloquium on Integrated Metallic Nanomaterials Systems. Hamburg, Germany. 30 Jun. 2021)

El-Zoka, A.: *Towards Enhanced Atom Probe Tomography Characterization of Wet Corrosion Systems* ((SCI Electrochemistry Postgraduate Conference. Manchester, UK. 11 Jun. 2021)

Freysoldt, C.; Hickel, T.; Janßen, J.; Wang, N.; Zendegani, A.: *High-throughput optimization of finite temperature phase stabilities: Concepts and application* (Coffee with Max Planck, Virtual Seminar organized by the MPIE. Düsseldorf, Germany. 04 Feb. 2021)

Gault, B.: *Advancing corrosion understanding with (cryo-) Atom Probe Tomography* (Imperial College London - Rolls Royce Corrosion Seminar, online. London, UK. 17 Jun. 2021)

Gault, B.: *An overview of the state-of-the-art of hydrogen analysis by APT* (APT school 2021, online. Rouen, France. 26 Oct. 2021)

Gault, B.: *Enabling Cryo-Atom Probe Tomography* (1st Grenoble Atom Probe Workshop, online. Grenoble, France. 16 Mar. 2021)

Gault, B.: *Introduction to atom probe tomography: performance and opportunities in characterizing microstructures* (Metallic Microstructures: European Lectures Online. 20 May 2021)

Gault, B.: *Machine-Learning for Atom Probe Tomography* (Workshop “Research-data management, machine learning and material informatics for Superalloys”, online. Bochum, Germany. 17 Jun. 2021)

Gault, B.: *Pushing the analytical limits of atom probe tomography via cryo-enabled workflows* (Microscience Microscopy Congress 2021, online. Oxford, UK. 08 Jul. 2021)

Gault, B.: *Pushing the limits of atomic-scale analytical microscopy* (University of Bristol Seminar, online. Bristol, UK. 01 Mar. 2021)

Gault, B.: *Theory and Advances in Atom Probe Tomography* (Canadian Centre for Electron Microscopy Summer School, online. Hamilton, ON, Canada. 08 Jun. 2021)

Gault, B.; Guillon, O.: *Du térawatt au picomètre: Voyage au cœur des technologies de l’hydrogène* (Café des Sciences de l’Ambassade de France en Allemagne, online. Berlin, Germany. 20 Jun. 2021)

Güder, Ü.: *Archaeological Traces of High Carbon Steel Metallurgy in Anatolia* (The Iranian Highlands: Resiliences and Integration in Premodern Societies, Deutsches Bergbau-Museum Bochum, online. Bochum, Germany. 20 Oct. 2021)

Güder, Ü.: *Following the fingerprints of ancient blacksmiths: reverse engineering of archaeological iron* (Pint of Science, online. Düsseldorf, Germany. 17 May 2021)

Güder, Ü.: *Historical and Technological Assessment of New Metals in Anatolia: Zinc in Urartian Bronze Alloys* (Seminar, Institute of Archaeology, Charles University, online. Prague, Czech Republic. 10 Nov. 2021)

Hickel, T.; Freysoldt, C.; Janßen, J.; Wang, N.; Zendegani, A.: *High-throughput optimization of finite temperature phase stabilities: Concepts and application* (Coffee with Max Planck, Virtual Seminar organized by the MPIE. Düsseldorf, Germany. 04 Feb. 2021)

Hickel, T.: *Application of Density Functional Theory in the Context of Phase Diagram Modelling* (MSIT Winter School on Materials Chemistry, Virtual Event. 02 May 2021)

Hickel, T.; Janßen, J.; Sözen, H. I.; Körmann, F.; Surendralal, S.; Todorova, M.; Neugebauer, J.: *High-throughput optimization of finite temperature phase stabilities: Concepts and application* (Thermec 2021, Virtual Conference. Graz, Austria. 11 May 2021)

Hickel, T.; Janßen, J.; Sözen, H. I.; Srrekala, L.; Körmann, F.; Surendralal, S.; Todorova, M.; Neugebauer, J.: *High-throughput optimization of finite temperature phase stabilities: Concepts and application* (ICAMS Advanced Discussions, virtual. Bochum, Germany. 08 Jun. 2021)

Huber, L.; Dsouza, R.; Poul, M.; Neugebauer, J.: *Defect Free Energies* (Interdisciplinary Centre for Advanced Materials Simulation (ICAMS) Seminar, delivered online. Bochum, Germany. 19 Jan. 2021)

Huber, L.: *Simulating the thermodynamics of GB phases* (Seminar, Institut für Materialswissenschaft (MaWi), University of Stuttgart, delivered online. Stuttgart, Germany. 08 Feb. 2021)

Janßen, J.; Hickel, T.; Neugebauer, J.: *pyiron – an integrated development environment for ab initio thermodynamics* (Potential Workshop, ICAMS, virtual. Bochum, Germany. 13 Mar. 2021)

Janßen, J.; Neugebauer, J.: *pyiron – an integrated development environment for material science* (CECAM Workshop, virtual. Lausanne, Switzerland. 25 Mar. 2021)

Khanchandani, H.; Stephenson, L.; Raabe, D.; Zaefferer, S.; Gault, B.: *Investigation of hydrogen embrittlement in high Mn TWIP steel via correlative microscopy (ECCI, APT)* (Cameca Online Webinar series for Atom Probe Tomography. Jul. 2021)

Kontis, P.; Antonov, S.; Kürnsteiner, P.; Katnagallu, S.; Mianroodi, J. R.; Lilensten, L.: *Development of superalloys driven by atomic-scale interactions of solutes with crystal defects* (TMS 2021, online. Pittsburgh, PA, USA. 15 Mar. 2021)

Liebscher, C.: *How do grain boundaries transform in metallic alloys?* (Institute of Material Physics, Westfälische Wilhelms-Universität Münster, Online Colloquium. Münster, Germany. 15 Jun. 2021)

Luo, T.; Gomell, L.: *Application of atom probe tomography in thermoelectrics research* (Cameca Online Webinar series for Atom Probe Tomography. Jun. 2021)

Lymperakis, L.: Nanomaterials from first principles (Seminar: Programme of Postgraduate course Materials Physics and Technology, Department of Physics, Aristotle University of Thessaloniki. Thessaloniki, Greece. 14 May 2021)

Meiners, T.; Frolov, T.; Duarte, M. J.; Richter, G.; Liebscher, C.; Dehm, G.: *Resolving grain boundary phase transformations in Copper by advanced STEM* (Thermec 2021, Online Conference. 01 Jun. 2021)

Neugebauer, J.: *Ab initio guided design of compositionally complex alloys* (Materials for energy, Digital Workshop. Bochum, Germany. 26 Feb. 2021)

Neugebauer, J.; Surendralal, S.; Deißenbeck, F.; Wippermann, S. M.; Freysoldt, C.; Todorova, M.: *Design and application of an ab initio electrochemical cell* (Spring Meeting of the German Physical Society (DPG), online. Berlin, Germany. 02 Mar. 2021)

Neugebauer, J.; Ikeda, Y.; Körmann, F.: *Materials design based on efficient sampling of high dimensional chemical and thermodynamic configuration spaces* (Workflows for Atomistic Simulations, Ruhr-Universität Bochum, Online Meeting. Bochum, Germany. 10 Mar. 2021)

Neugebauer, J.; Janßen, J.: *Pyiron – an integrated development environment for simulation workflows* (CECAM Summer School in 2020: “Simulation Workflows in Materials Modelling (SWiMM 2020)”, CECAM HQ – École Polytechnique Fédérale de Lausanne, Virtual Meeting. Lausanne, Switzerland. 23 Mar. 2021)

Neugebauer, J.: *Materials design by exploiting high-dimensional chemical and structural configuration spaces* (Kolloquium im Rahmen des SFB 986, Technische Universität Hamburg-Harburg, Online Meeting. Hamburg-Harburg, Germany. 26 May 2021)

Neugebauer, J.; Zendegani, A.; Hickel, T.: *Defect phase diagrams as novel tool to understand and design tailored defect structures in advanced steels* (Thermec 2021, Virtual Meeting. Vienna, Austria. 03 Jun. 2021)

Neugebauer, J.: *First principles first* (ICAMS Advanced Discussions 2021: Recent trends in materials design, Virtual Conference. Bochum, Germany. 08 Jun. 2021)

Neugebauer, J.: *Panel discussion leader* (ICAMS Advanced Discussions 2021: Recent trends in materials design, Virtual Conference. Bochum, Germany. 08 Jun. 2021)

Neugebauer, J.: *Efficient sampling of high-dimensional chemical and thermodynamic configuration spaces* (ELRC2020: Invitation to Complex High-Dimensional Energy Landscapes Reunion Conference II, delivered online. Lake Arrowhead, CA, USA. 10 Jun. 2021)

Neugebauer, J.: *Ab initio descriptors to guide materials design in high-dimensional chemical and structural configuration spaces* (Münchner Physik Kolloquium - Festkolloquium für Professor Winfried Petry, Technische Universität München, delivered online. München, Germany. 21 Jun. 2021)

Neugebauer, J.; Surendralal, S.; Deißenbeck, F.; Wippermann, S. M.; Todorova, M.: *Identifying and understanding corrosion reactions: An ab initio approach* (ICASS - 4th International Conference on Applied Surface Science, Virtual Conference. 30 Jun. 2021)

Neugebauer, J.: Ab initio descriptors to guide materials design in high-dimensional chemical and structural configuration spaces (EPFL Materials Science and Engineering (IMX) Seminar Series, virtual. Lausanne, Switzerland. 22 Nov. 2021)

Neugebauer, J.; Yoo, S.-H.; Lymperakis, L.: *Ab initio insights into fundamental intrinsic growth and materials limitations in group-III-nitrides* (MRS Fall Meeting, Virtual Conference, virtual. Boston, MA, USA. 28 Nov. 2021)

Palm, M.: *Phase Equilibria and Phase Diagrams* (5th MSIT Winter School on Materials Chemistry. Online. 3 May 2021)

Palm, M.: *Iron aluminides – a class of sustainable materials* (Intermetallics 2021, Educational Center Kloster Banz, Germany. 7 Oct. 2021)

Peter, N. J.: *Chemically triggered faceting transition of an asymetric tilt grain boundary in Cu* (Department of Metallurgical Engineering and Materials Science, Indian Institute of Technology, virtual. Bombay, India. 28 Jul. 2021)

Raabe, D.: *The Science of dirty alloys: recycling-based Aluminium for a circular economy* (The 4th International Conference on Light Materials - Science and Technology, online. 02 Nov. 2021)

Raabe, D.; Kim, S.-H.; Zhang, X.; Ma, Y.; Souza Filho, I. R.; Schweinar, K.; Angenendt, K.; Vogel, D.; Stephenson, L.; El-Zoka, A.; Mianroodi, J. R.; Rohwerder, M.; Gault, B.; Bai, Y.; Springer, H.; Ponge, D.; Kulse, M.; Mahajan, A.; Zaefferer, S.: *Sustainable Metals* (Virtual Keynote Perspective Lecture, Materials Chain and Materials Science Department, RUB. Bochum, Germany. 07 May 2021)

Raabe, D.; Ponge, D.; Kwiatkowski da Silva, A.; Makineni, S. K.; Katnagallu, S.; Zhou, X.; Kamachali, R. D.; Stephenson, L.; Kontis, P.; Mianroodi, J. R.; Gault, B.; Thompson, G. B.; Neugebauer, J.: *Structure, chemistry and thermodynamics of Lattice Defects* (Colloquium of the Department of Materials Science and Engineering, Ohio State Universitry, online. Columbus, OH, USA. 26 Mar. 2021)

Ramachandramoorthy, R.: *High strain rate testing from micro-to-meso scale* (MRS Spring Meeting - In Situ Mechanical Testing of Materials at Small Length Scales, Modeling and Data Analysis Symposium, online. 17 Apr. 2021)

Ramachandramoorthy, R.: *High strain rate testing of copper based micropillars and microlattices* (206 Departmental Seminar Series, Empa. Thun, Switzerland. 22 Jun. 2021)

Ramachandramoorthy, R.: *Micro/nanoscale fabrication techniques* (Small scale mechanics course, RUB. Bochum, Germany. 17 May 2021)

Ramachandramoorthy, R.: *Pushing the limits of microscale manufacturing and mechanical testing* (Department of Material Science and Engineering Seminar Series, Tel-Aviv University, online. Tel-Aviv, Israel. 18 May 2021)

Ramachandramoorthy, R.: *Small scale mechanical characterization* (Small scale mechanics course, RUB. Bochum, Germany. 31 May 2021)

Rohwerder, M.: *Scanning Kelvin Probe Techniques for the Investigation of Corrosion, Hydrogen Uptake and Permeation of Zinc Alloy Coatings on Steels* (Galvatech 2021, Virtual Conference. 21 Jun. 2021)

Roters, F.; Diehl, M.; Eisenlohr, P.; Shanthraj, P.: DAMASK: the Düsseldorf Advanced MAterial Simulation Kit for studying multi-field crystal plasticity phenomena (Seminar, Engineering Science Department at the University of Oxford, virtual. Oxford, UK. 18 Oct. 2021)

Scheu, C.; Gleich, S.; Soler, R.; Breitbach, B.; Bolvardi, H.; Achenbach, J.-O.; Schneider, J. M.; Dehm, G.: *Insights in the Structure, Defects and Stability of* Mo_2BC *Thin Films by Advanced Characterization Methods* (International Conference on Metallurgical Coatings and Thin Films 2021 (ICMCTF2021), Virtual Conference. 26 Apr. 2021)

C. Scheu: *Tracing impurities at surfaces and interfaces of renewable energy materials* (MS&T 21, Online Conference, Columbus, Ohio. 17 Oct. 2021)

Scheu, C.; Hengge, K. A.: *Insights in the stability of Pt/Ru catalyst and the effect for polymer electrolyte membrane fuel cells* (Thermec 2021, Online Conference. 01 Jun. 2021)

Scheu, C.; Zhang, S.: *Hematite for light induced water splitting – improving efficiency by tuning distribution of Sn dopants at the atomic scale* (Karlsruher Werkstoffkolloquium_Digital. 15 Jun. 2021)

Siemer, N.: *Metadata-schemes and RDM handbook in SFB1394* (Workshop “Research-data management, machine learning and material informatics within the SFB/TR103”, RUB, virtual. Bochum, Germany. 10 Jun. 2021)

Stein, F.: *Experimental Determination of Phase Diagrams* (Lecture, 5th MSIT Winter School on Materials Chemistry, virtual. 02 May 2021)

Surendralal, S.; Todorova, M.: *Corrosion at the Quantum Level* (Coffee with Max Planck, Virtual Seminar organized by the MPIE. Düsseldorf, Germany. 04 Feb. 2021)

Todorova, M.; Surendralal, S.: *Corrosion at the Quantum Level* (Coffee with Max Planck, Virtual Seminar organized by the MPIE. Düsseldorf, Germany. 04 Feb. 2021)

Todorova, M.; Surendralal, S.; Wippermann, S. M.; Deißenbeck, F.; Freysoldt, C.; Neugebauer, J.: *Insights into processes at electrochemical solid/liquid interfaces from ab initio molecular dynamics simulations* (TMS 2021, online. Orlando, FL, USA. 16 Mar. 2021)

Todorova, M.; Surendralal, S.; Wippermann, S. M.; Deißenbeck, F.; Neugebauer, J.: *Hydrogen at electrified solid/liquid interfaces – insights from ab initio molecular dynamics simulations* (TYC mini-workshop on “Physics and Chemistry of Solid/Liquid Interfaces”, online. London, UK. 20 May 2021)

Todorova, M.; Surendralal, S.; Wippermann, S. M.; Deißenbeck, F.; Neugebauer, J.: *Insights into processes at electrochemical solid/liquid interfaces from ab initio molecular dynamics simulations* (ICTP-Workshop on “Physics and Chemistry of Solid/Liquid Interfaces for Energy Conversion and Storage”, Virtual Meeting. Trieste, Italy. 28 May 2021)

Todorova, M.; Yoo, S.-H.; Vatti, A. K.; Neugebauer, J.: *From Semiconductor defect chemistry to electrochemistry* (ICACES/BENCh Virtual Summer School 2021. Göttingen, Germany. 14 Jun. 2021)

Todorova, M.; Surendralal, S.; Wippermann, S. M.; Deißenbeck, F.; Neugebauer, J.: *Insights into processes at electrochemical solid/liquid interfaces from ab initio molecular dynamics simulations* (ICACES/BENCh Virtual Summer School 2021. Göttingen, Germany. 14 Jun. 2021)

Todorova, M.; Surendralal, S.; Wippermann, S. M.; Deißenbeck, F.; Neugebauer, J.: *Processes at solid/liquid interfaces – insights from ab initio molecular dynamics simulations with potential control* (AMaSiS 2021 Online - Applied Mathematics and Simulation for Semiconductors and Electrochemical Systems. Berlin, Germany. 06 Sep. 2021)

Todorova, M.; Surendralal, S.; Wippermann, S. M.; Deißenbeck, F.; Neugebauer, J.: *Realistic description of processes at solid/liquid interfaces by ab initio molecular dynamics simulations with potential control* (MRS Fall Meeting. Boston, MA, USA. 7 Dec. 2021)

Tsai, S.-P.; Zaefferer, S.: *Large-volume 3D EBSD system and its application to the investigation of grain boundary corrosion in 316L stainless steel* (3D MS conference, online. 30 Jun. 2021)

Yoo, S.-H.: *Electronic passivation schemes for surfaces with spontaneous polarization and for low symmetry semiconductor surfaces in DFT slab calculations* (Materials Design Group Seminar, Department of Materials, Imperial College London, virtual. London, UK. 26 Apr. 2021)

Yoo, S.-H.: *Overcoming failing Size Convergence for Surface Calculations of Materials Exhibiting Spontaneous Polarization* (International Conference Materials Science and Engineering: Materials Oceania 2021, virtual. Brisbane City, Australia. 13 Oct. 2021)

Zaefferer, S.: *2D and 3D SEM-based electron diffraction techniques as central tools for correlative microscopy to obtain new insights into microstructure physics and chemistry* (Kolloquium des Zentrums für Elektronenmikroskopie, online. Graz, Österreich. 09 Mar. 2021)

Zaefferer, S.: *Towards understanding hydrogen embrittlement: Tools for microscopic and nanoscopic detection of hydrogen and its mechanical effects in microstructures of steels and superalloys* (Colloquium of the Institute of Materials Science, TU Freiberg, online. Freiberg, Germany. 10 May 2021)

Publications (as of November 2021)

A comprehensive listing of scientific publications is available on the following institute's web pages:

Computational Materials Design:
https://www.mpie.de/2967008/publications

Interface Chemistry and Surface Engineering:
https://www.mpie.de/2999511/Interface_Chemistry_and_Surface_Engineering

Microstructure Physics and Alloy Design:
https://www.mpie.de/2987664/Publications

Structure and Nano-/ Micromechanics of Materials:
https://www.mpie.de/2999835/publications

Nanoanalytics and Interfaces:
https://www.mpie.de/3118894/Nanoanalytics_and_Interfaces

Open Access:
https://www.mpie.de/4263389/open-access-publications

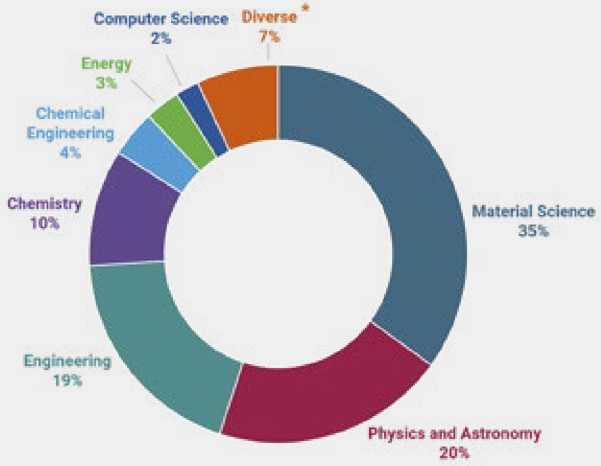
Publication statistics

The results of the fundamental scientific research conducted at the MPIE are mainly published in peer-reviewed scientific journals. The following publication statistics are extracted from SciVal, which is based on the Scopus database of indexed scientific journals. [1]

From January 2019 to November 2021, **831** peer-reviewed journal articles and reviews were published by the institute; over **50%** of the total set are **Open Access** and hence made broadly accessible across the scientific community but also the general public, with a mix between Gold and Green as detailed below.

Publications by subject area

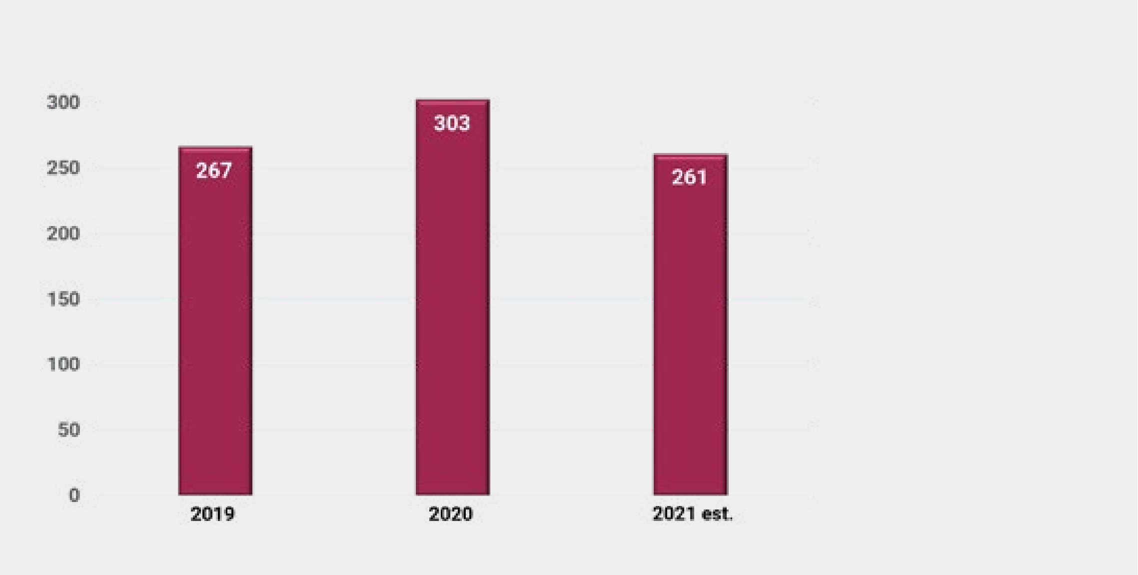
High level subject areas for journals have been defined by the All Science Journal Classification (ASJC), and each journal and article therein can hence belong to more than one ASJC.[1]
The set of publications between 2019 - 2021 from the MPIE cover a broad range of topics, centred on materials science & engineering and physics, but also with forays into chemistry, energy and computer science.



*Diverse includes Biochemistry, Genetics and Molecular Biology/ Decision Sciences/ Dentistry/ Earth and Planetary Sciences/ Environmental Science/ Health Professions/ Mathematics/ Medicine/ Pharmacology, Toxicology and Pharmaceutics and Social Sciences.

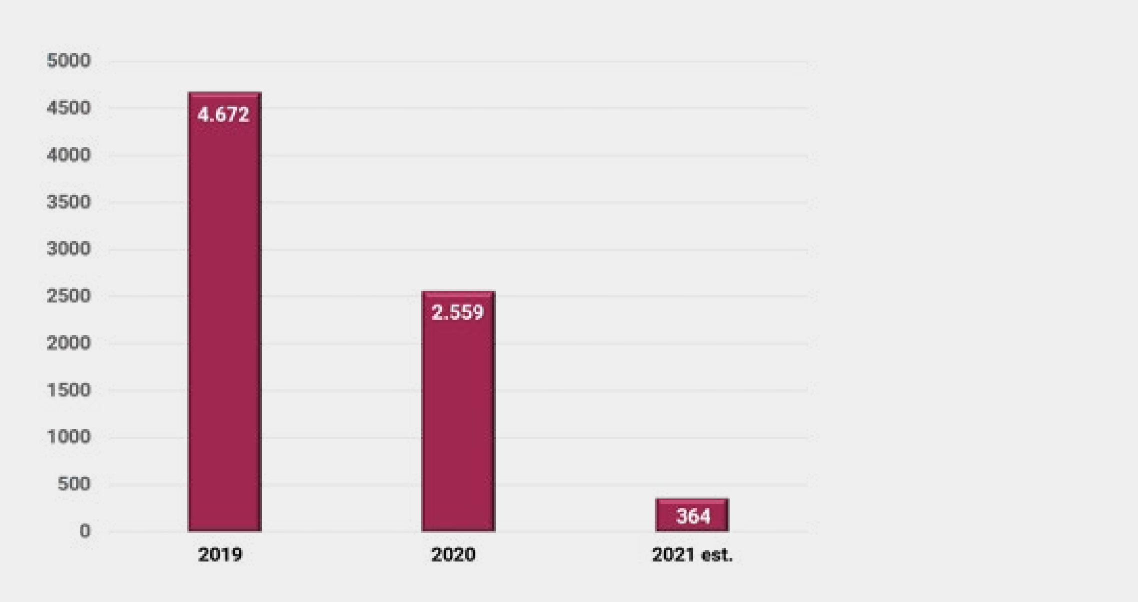
Scholarly output

A total of **831** publications was released until 24 November 2021.



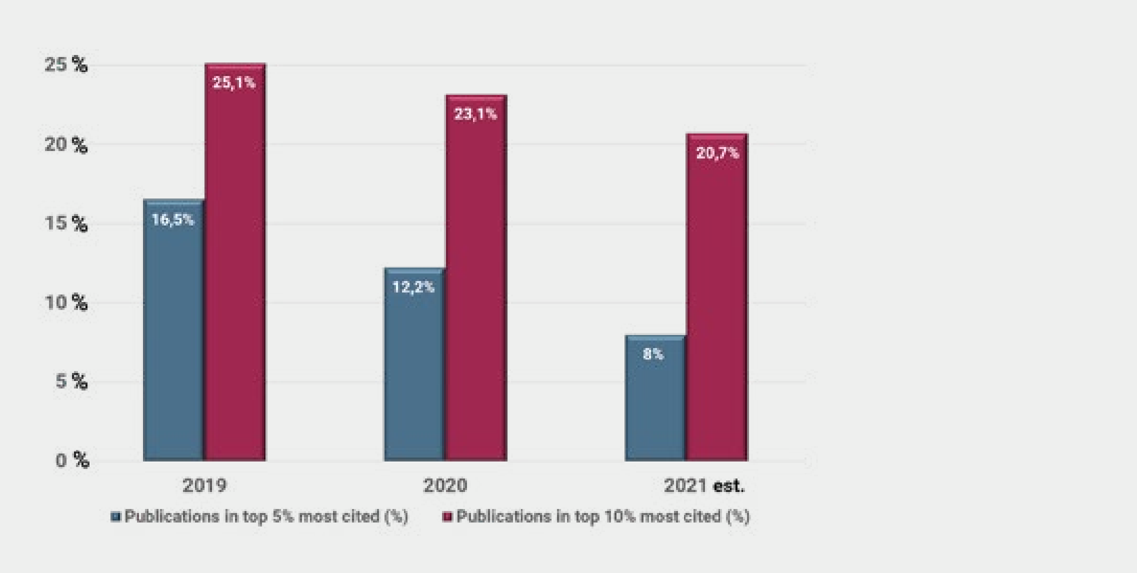
Citation count

A total of **7.595** citations was received by publications at the MPIE between 2019 – 2021 with **an average of 9.3** citations per publication. The citation count indicates how many citations these publications received. The years are always the years in which items were published, and do not refer to the years in which citations were received.



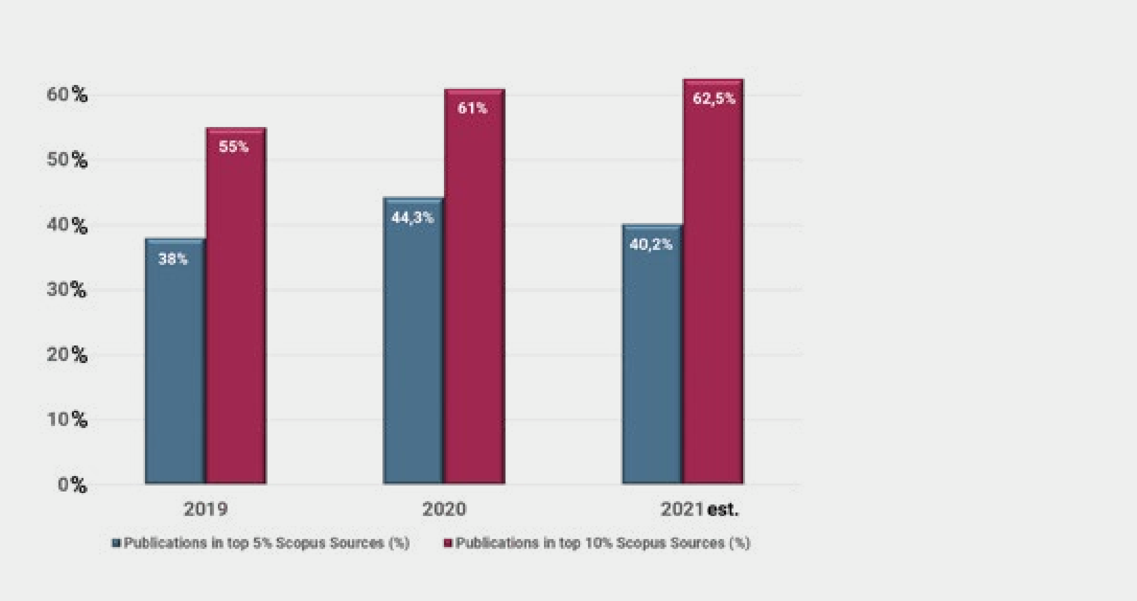
This is the total citations for the publication set, with years being are the years when articles were published, not to the years in which citations were received. [1]

189 (23%) publications are **in the top 10% most cited publications** worldwide in their respective field.



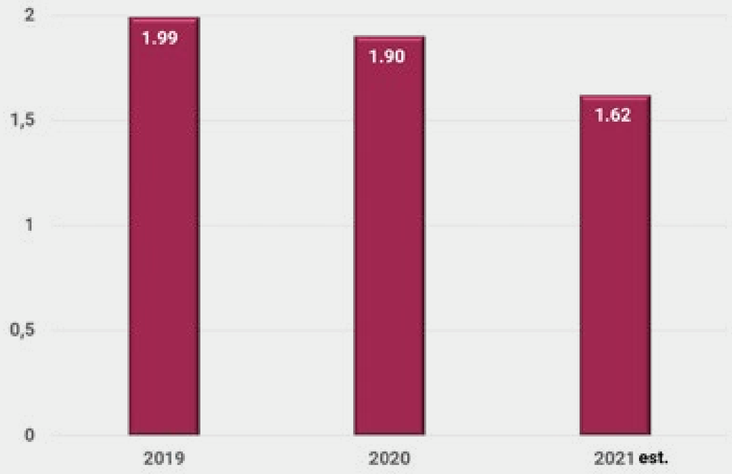
482 (59,6%) publications are **in the top 10% journals** by CiteScore.

These values indicate the extent to which the set of publication from the MPIE is present in the most-cited journal articles referenced in Scopus, and presented by CiteScore percentile.[1]



The field-weighted citation **impact** of publications at the MPIE is **1.85**.

The field-weighted citation impact (FWCI) indicates how the number of citations received by the considered set of publications compares with the average number of citations received by all other publications indexed in Scopus in similar categories. A FWCI of 1.00 indicates that the set has been cited exactly as would be expected based on the global average for similar publications across the considered field. A FWCI of more than 1.00 indicates above average citations.[1]



Scholarly output in publications by amount of international, national and institutional collaboration

	Scholarly output	Citations	Citations per publication	Field-weighted citation impact
International collaboration	621	6.051	9,7	1,89
Only national collaboration	135	827	6,1	1,49
Only institutional collaboration	56	589	10,5	2,14
Single authorship (no collaboration)	9	128	14,2	2,34

"The scholarly output in publications by amount of international, national and institutional collaboration indicates the extent to which the publication set's publications have international, national, or institutional co-authorship, and single authorship. A publication is assigned a single collaboration type."[1]

Institutions participating in MPIE publications

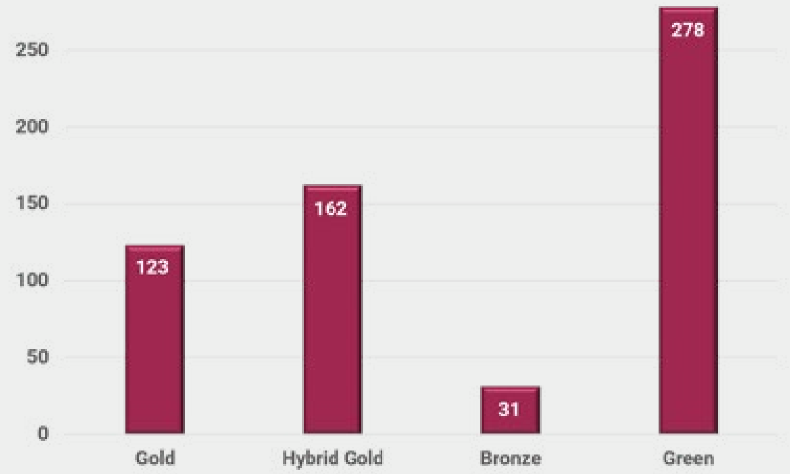
Name	Scholarly output	Authors	Citations per publication
1. MPIE	821	344	9,3
2. Imperial College London	121	16	7,8
3. RWTH Aachen University	109	95	7,9
4. Central South University	62	46	10,0
5. Ruhr University Bochum	50	64	10,8
6. CNRS	47	80	6,5
7. Delft University of Technology	46	31	15,0
8. Jülich Research Centre	37	26	10,1
9. University of Science and Technology Beijing	36	69	7,2
10. Friedrich-Alexander University Erlangen- Nürnberg	31	24	13,1

Selected journals

Name	Number of publications
Acta Materialia	111
Scripta Materialia	56
Materials Science And Engineering A	40
Nature Communications	23
Physical Review B	14
Physical Review Letters	8
Advanced Materials	7
Science Advances	6
Nature	4
Nature Materials	4

Open access

A total of **419** papers was published open access between 2019 and 2021.
A list of our open access publications is available on our webpage:
https://www.mpie.de/4263389/open-access-publications



“Gold
Documents that are in journals which only publish open access.

Hybrid gold
Documents that are in journals which provide authors the choice of publishing open access.

Bronze
Published version of record or manuscript accepted for publication. The publisher has chosen to provide temporary or permanent free access.

Green
Published version or manuscript accepted for publication, available at repository.” [1]

References

1. SciVal, Publication Set Overview Report prepared on the 4th of November 2021.

Habilitation, doctoral, master and bachelor theses

Habilitation theses

2019

PD Dr. S. Brinckmann: Exploration of Crack Propagation in Metals by Length-Scale Dependent Simulations (Ruhr-Universität Bochum)

2021

PD Dr. M. Herbig: Hüftimplantate: Ein werkstoffwissenschaftlicher Blick auf Geschichte, Möglichkeiten und Limitationen (RWTH Aachen)

Doctoral theses

2018 (not included in Scientific Report 2016 - 2018)

Dr. rer. nat. M. Sysoltseva: Characterization of aerosols and nanoparticles released during various indoor and outdoor human activities (RWTH Aachen)

2019

Dr.-Ing. L. Abdellaoui: Correlation of microstructures and thermal conductivity of the thermoelectric material $Ag_{16.7}Sb_{30}Te_{53.3}$ (Ruhr-Universität Bochum)

Dr.-Ing. D. An: A study on low-cycle fatigue behaviour of high-manganese steels (RWTH Aachen)

Dr.-Ing. Y. Chang: Challenges and opportunities associated to the characterization of H/D in Ti and its alloys with atom probe tomography (RWTH Aachen)

Dr.-Ing. A. Dutta: Deformation behaviour and texture memory effect of multiphase nano-laminate medium manganese steels (RWTH Aachen)

Dr.-Ing. A. Gupta: Ab initio based study of precipitate formation in advanced structural Al-based alloys (Ruhr-Universität Bochum), **with distinction ”summa cum laude”**

Dr.-Ing. A. Hariharan: On the interfacial defect formation mechanism during laser additive manufacturing of polycrystalline superalloys (Ruhr-Universität Bochum)

Dr.-Ing. P. Kerger: Absolute Electrode Potentials - The Thin Film Electrode (Ruhr-Universität Bochum)

Dr.-Ing. A. Kini: Laser Additive Manufacturing of Oxide Dispersion Strengthened Steels and Cu−Cr−Nb Alloys (RWTH Aachen)

Dr.-Ing. D. Korbmacher: Ab initio study of strongly anharmonic and dynamically unstable systems (Ruhr-Universität Bochum)

Dr. rer. nat. P. Kürnsteiner: Precipitation Reactions During the Intrinsic Heat Treatment of Laser Additive Manufacturing (RWTH Aachen), **with distinction ”summa cum laude”**

Dr.-Ing. W. Luo: Mechanical properties of the cubic and hexagonal $NbCo_2$ Laves phases studied by micromechanical testing (Ruhr-Universität Bochum)

Dr.-Ing. T. Meiners: Grain Boundary Structure, Phase Transitions and Segregation Phenomena in Copper Alloys (Ruhr-Universität Bochum)

Dr.-Ing. A. Merz: Investigation of the “Protection Zone”, a novel mechanism to inhibit delamination of composite organic coatings containing conducting polymer (Ruhr-Universität Bochum)

Dr.-Ing. H.I. Sözen: Ab initio phase stabilities of Ce-based hard magnetic materials (Ruhr-Universität Bochum)

Dr. rer. nat. S. Tecklenburg: Defect formation and evolution in zinc oxide: from semiconductors to corrosion (Ruhr-Universität Bochum)

Dr.-Ing. M. Uebel: Release and transport of corrosion inhibitors in self-healing coatings for intelligent corrosion protection (Ruhr-Universität Bochum), **with distinction ”summa cum laude”**

Dr.-Ing. D. Wang: Damage and Strain Patterning Simulation of Structural Heterogeneity (RWTH Aachen)

Dr.-Ing. C.-H. Wu: The Principle and Applications of Scanning Kelvin Probe based Hydrogen Detection Technique on Pd-coated and Oxide Covered Surface (Ruhr-Universität Bochum)

Dr.-Ing. H. Zhao: Segregation and precipitation at interfaces in a model Al−Zn−Mg−Cu alloy (RWTH Aachen)

2020

Dr.-Ing. V.G. Arigela: Development and application of a high-temperature micromechanics stage with a novel temperature measurement approach (Ruhr-Universität Bochum)

Dr.-Ing. P. Bajaj: Precipitation during Intrinsic Heat Treatment in Laser Additive Manufacturing (RWTH Aachen)

Dr.-Ing. C. Corrêa da Silva: Investigation of the kinetics of selective oxidation of iron model alloys during simulated (Ruhr-Universität Bochum)

Dr.-Ing. P. Keuter: Design of materials with anomalous thermophysical properties and desorption-assisted phase formation of intermetallic thin films (RWTH Aachen University), **with distinction ”summa cum laude”**

Dr.-Ing. J. Li: Probing dislocation nucleation in grains and at $\Sigma3$ twin boundaries of Cu alloys by nanoindentation (Ruhr-Universität Bochum)

Dr.-Ing. S. Nandy: Investigations on the Mechanisms of Ductilization of Mg Alloys with Ca, Al and Zn (Ruhr-Universität Bochum)

Dr.-Ing. R. Pei: Microstructural Relationships of Strength and Ductility in a Newly Developed Mg–Al–Zn Alloy for Po-tential Automotive Applications (RWTH Aachen)

Dr.-Ing. Y. Qin: Microstructure alterations in bearing steels exposed to severe plastic deformation (RWTH Aachen)

Dr. M. Schestakow: Nanostrukturierte Cellulose-Aerogel-Polyesterverbunde (RWTH Aachen University), **with distinction ”summa cum laude”**

Dr.-Ing. S. Surendralal: Development of an ab initio computational potentiostat and its application to the study of Mg corrosion (Ruhr Universität Bochum), **with distinction ”summa cum laude”**

Dr.-Ing. S.-H. Yoo: Development of a computational framework to determine the chemical and thermodynamic stability of electrode materials in an electrochemical environment (Ruhr-Universität Bochum)

Dr.-Ing. A. Zendegani: Ab initio based study of the thermodynamics of complex intermetallics (Ruhr-Universität Bochum)

2021

Dr. rer. Nat. J. Janßen: pyiron – an Integrated Development environment for ab initio Thermodynamics (Paderborn University), **with distinction ”summa cum laude”**

Dr.-Ing. S.-H. Kim: Engineering Impurities in Colloidal Nanostructures used in “Green Hydrogen” Generation (RWTH Aachen University), **with distinction ”summa cum laude”**

Dr. rer. Nat. F. Lochner: Interplay of Real Space and Electronic Structure for Iron-Based Superconductors: An ab initio Study (Ruhr-University-Bochum)

Dr. D. Mayweg: Microstructural characterization of white etching cracks (RWTH Aachen)

Dr.-Ing. S.A.H. Motaman: Modeling of the microstructural effects on the mechanical response of poly-crystals (RWTH Aachen)

Dr. M. Obermaier: Development of characterization methods to improve automotive PEMFC degradation analysis (RWTH Aachen)

Dr.-Ing. N.J. Peter: Structure, chemistry and nanomechanics of grain boundaries in Cu–Ag alloys (Ruhr-Universität Bochum)

Dr. Z. Rao: Design of Invar and Magnetic High-Entropy Alloys (RWTH Aachen)

Dr.-Ing. K. Schweinar: Advancements in the understanding of Ir-based water splitting catalysts at the near-atomic scale (Ruhr-Universität Bochum), **with distinction ”summa cum laude”**

Dr. mont. L. Stemper: Crossover-alloys – a new approach for future aluminum alloys (University Leoben, Austria), **with distinction ”summa cum laude”**

Dr.-Ing. C. Tian: On the damage initiation in dual phase steels: Quantitative insights from in situ micromechanics (Ruhr-Universität Bochum)

Dr.-Ing. P.-Y. Tung: Fundamentals of carbide decomposition and stability (Ruhr-Universität Bochum)

Dr.-Ing. S. Vakili: Multi-Phase-Field Modeling of Structure Formation in Metallic Foams (Ruhr-Universität Bochum)

Dr.-Ing. R.S. Varanasi: Mechanisms of refinement and deformation of novel ultrafine-grained medium manganese steels with improved mechanical properties (Ruhr-Universität Bochum)

Dr. rer. nat. Y. Wei: Machine learning methods in data-driven nanostructure analysis of materials (RWTH Aachen)

Dr.-Ing. S. Wolff-Goodrich: Development of AlCrFeNiTi Compositionally Complex Alloys for High Temperature Structural Applications (Ruhr-Universität Bochum)

Master theses

2019

R. Aymerich Armengol: Structure-property relationship studies of Pt/TiO_2 nanomaterials for electrochemical applications (Universitat Autònoma de Barcelona, Spain)

R. Bueno Villoro: Effect of the processing route on the microstructure of $Ag_{18}Sb_{29}Te_{53}$ (AST) based thermoelectrics (Universitat Autònoma de Barcelona, Spain)

R. Dsouza: Fully anharmonic self-diffusion coefficients using the Finite-Temperature String method (Ruhr-Universität Bochum)

F. Farzam: Microstructure Evolution in FCC Metals during Friction (Heinrich-Heine-Universität Düsseldorf)

U. Gajera: Phase diagrams derived from optimized empirical potentials (Ruhr-Universität Bochum)

D.R. Machado: Investigation of the low cyclic fatigue behavior of high manganese steels (RWTH Aachen)

S. Roongta: A Physics Based Crystal Plasticity Model for Cyclic Deformation (Ruhr-Universität Bochum)

H.C. Sam: Role of microstructure and environment on delayed fracture in a novel lightweight medium manganese steel (Augsburg University)

2020

A. Aslam: Thermodynamics of binary alloys at atomistic scale (Ruhr-Universität Bochum)

X. Dong: Hydrogen Associated Decohesion and Localized Plasticity in a Dual-phase Lightweight Steel (RWTH Aachen)

N. Kusampudi: Using Machine Learning and Data-driven Approaches to Predict Damage Initiation in Dual-Phase Steels (Ruhr-Universität Bochum)

L. Lahn: On the role of defects in Ir-Ru alloys for energy conversion applications (RWTH Aachen)

A. Saxena: Machine learning the formation of defect phases in aluminium alloys (Ruhr-Universität Bochum)

C.K. Soundararajan: Recrystallization behavior and mechanical properties of interstitially alloyed CoCrFeMnNi equiatomic high entropy alloy (RWTH Aachen University)

2021

Ç. Aras: Anharmonic free-energy calculation of dynamically unstable crystal structures (RWTH Aachen)

V. Bhuva: Algorithms for optimal chemical ordering (University of Passau)

B. Hill: Scanning electron microscopy study of the microscale degradation mechanisms in polymer electrolyte fuel cells (Heinrich-Heine-Universität Düsseldorf)

A. Kokot: Solidification and precipitated structure of $Al_{20}Co_xCr_{20}Fe_{35}Ni_{20-x}Ti_5$ (x = 0, 10, 20) compositionally complex alloys (Heinrich-Heine-Universität Düsseldorf)

P. Mathews: Concentration-dependent finite temperature effects in metallic alloys (Ruhr-Universität Bochum)

A.W. Paiva do Nascimento: An optimized method to determine initial parameters of advanced yield surfaces for sheet metal form-ing applications (Ruhr-Universität Bochum)

M. Vega Paredes: Electron microscopy degradation studies of ruthenium-platinum core-shell nanoparticles for polymer electrolyte membrane fuel cells (Universitat Autònoma de Barcelona, Spain)

Bachelor theses

2020

M. Vega Paredes: Degradation mechanisms during operation of high temperature polymer electrolyte membrane fuel cells (Universitat Autònoma de Barcelona, Spain)

Scientific community service activities of the directors

Prof. Gerhard Dehm

Member of the Selection Committee for Applicants for Max-Planck-Lise-Meitner Groups (since 2018)

Member of the Selection Committee for the Allocation of Humboldt Research Fellowships (since 2017)

Member of the Selection Committee for Applicants for Max-Planck-Research Groups (since 2014)

Member of the board of trustees (Kuratorium) of the KSB Stiftung (since 2016)

Member of the Board of Governors of Acta Materialia Inc. (since 2013)

Editorial Board of Advanced Engineering Materials (Wiley) (since 2013)

Member of the Editorial Board International Journal of Materials Research (Hanser) (since 2007)

Member of the Editorial Board Berg- und Hußttenmannische Monatshefte (Springer) (since 2009)

Prof. Jörg Neugebauer

Member (since 2004) and speaker (since 2014) of the International Max Planck Research School for Interface Controlled Materials for Energy Conversion (IMPRS-SurMat) at the Max-Planck-Institut für Eisenforschung, the Ruhr-Universität Bochum, the University of Duisburg-Essen, the Max-Planck Institut für Kohlenforschung and the Max-Planck-Institute for Chemical Energy Conversion

Member of the IT-advisory board (BAR) of the Max-Planck-Society (since 2006)

Director of the Advanced Study Group “Modelling” at the International Centre of Advanced Materials Simulation ICAMS (since 2008)

Member of the Scientific Committee of the PSI-k charity and Speaker of the Working Group Applications to real materials: Structural materials (since 2009)

Elected Member of the Academy of Sciences and Art in North Rhine-Westphalia (since 2010)

Elected chair of the Metals and Materials (MM) Division of the German Physical Society (DPG) (2016 - 2019)

Elected member of the working group Metals and Materials (AGMM) of the German Physical Society (DPG) (since 2019)

Elected member of the DFG review board (DFG-Fachkollegium) „Materials Science“ (2016 - 2020)

Member of the HLRB (supercomputer in Bavaria) steering committee of the Leibniz computer centre of the Bavarian Academy of Sciences (since 2012)

Foreign Member of the Austrian Academy of Sciences (since 2012)

Chair of the expert committee “Digital transformation in materials engineering” of the Association of German Engineers (VDI) (since 2016)

Associate Editor, npj Computational Materials (since 2015)

Member of the scientific advisory board of the Physics Department of the University of Jena (2015 - 2020)

Member of the scientific advisory board GraFOx, Graduate school of several Leibnitz institutes in Berlin

Member of the Management Board of the Consortium Material Digital (since 2019)

Member of the Supervisory Board (Aufsichtsrat) of the Karlsruhe Institute of Technology (KIT) (since 2019)

Member of the scientific committee of the “Models and Data for Plasma-Material Interaction in Fusion Devices (MoD-PMI)” workshop series (since 2016)

Member of the High Entropy Alloys (HEA 2021) Conference International Advisory Committee (since 2021)

Prof. Dierk Raabe

Member of the Selection Board of the scholarship programme of the Alexander von Humboldt Foundation since 2007

Member of selection board of the Alexander von Humboldt Professorship programme (since 2017)

Member of the Governors Board of Acta Materialia Inc. (2007-2015)

Chairman of the RWTH Board of Governors (Hochschulrat der RWTH Aachen) (2012-2017)

Member of the Council of Science and Humanities of the German Government (Wissenschaftsrat) (2010- 2016)

Senator of the Helmholtz Association for the Research Field Key Technologies (since 2017)

Member Northrhine-Westfalian Academy of Sciences since 2008

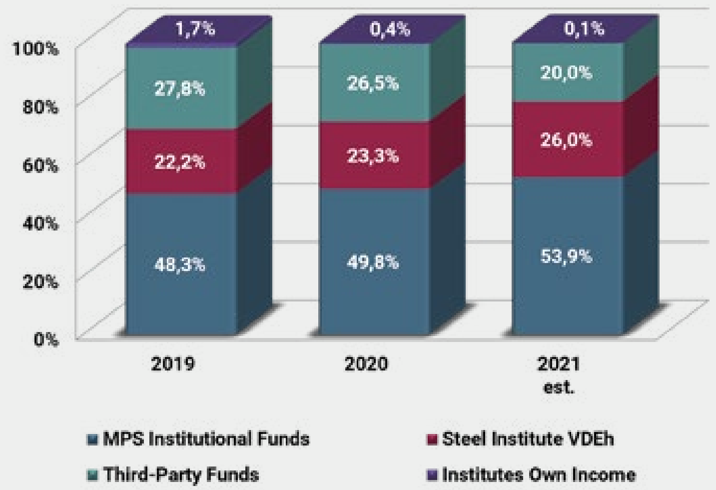
Member National Academy Leopoldina, 2013

Honorary Professor at Katholieke Universiteit Leuven, Belgium, 2014

Budget of the institute

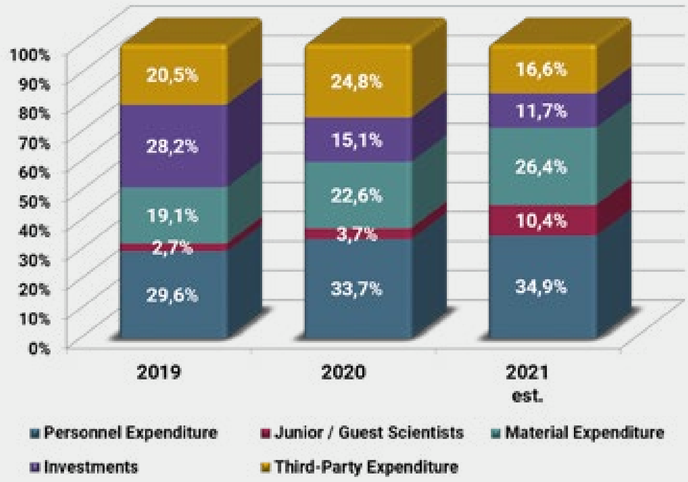
Revenue

(Percentual contributions to total revenue without appointment-related investment funds and general reconstruction of the buildings; year 2021 data estimated)



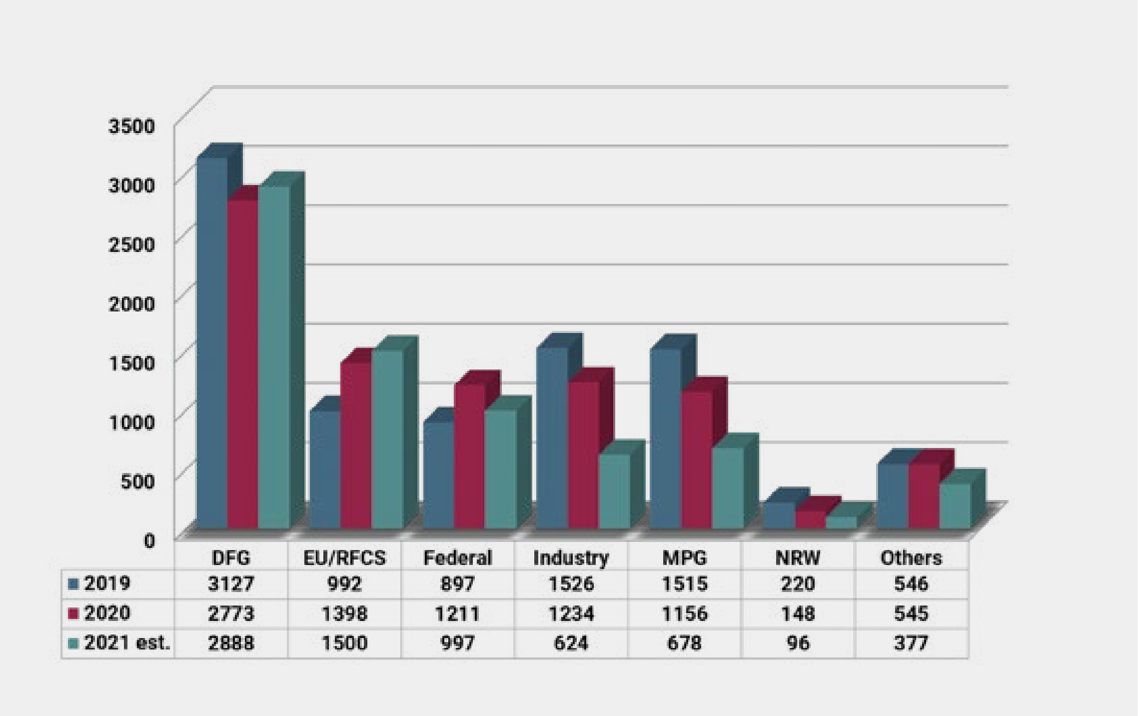
Expenditure

(Percentual distribution of total expenditure; investments include large-scale apparatus, electronic data processing, appointment-related investments, separate investment for basic equipment; year 2021 data estimated)



Third-party funds

(Contributions in 1,000 € to total revenue including personnel, material and investements, year 2021 data estimated)



	DFG	EU/RFCS	Federal	Industry	MPG	NRW	Others
🟦 2019	3127	992	897	1526	1515	220	546
🟥 2020	2773	1398	1211	1234	1156	148	545
🟩 2021 est.	2888	1500	997	624	678	96	377

DFG: German Science Foundation

EU: European Union

RFCS: Research Fund for Coal and Steel

Federal: BMBF & BMWi

BMBF: Federal Ministry of Science and Education

BMWi: Federal Ministry of Economics and Technology

Industry incl. Christian Doppler Society and MaxNet

MPG: Max Planck Society

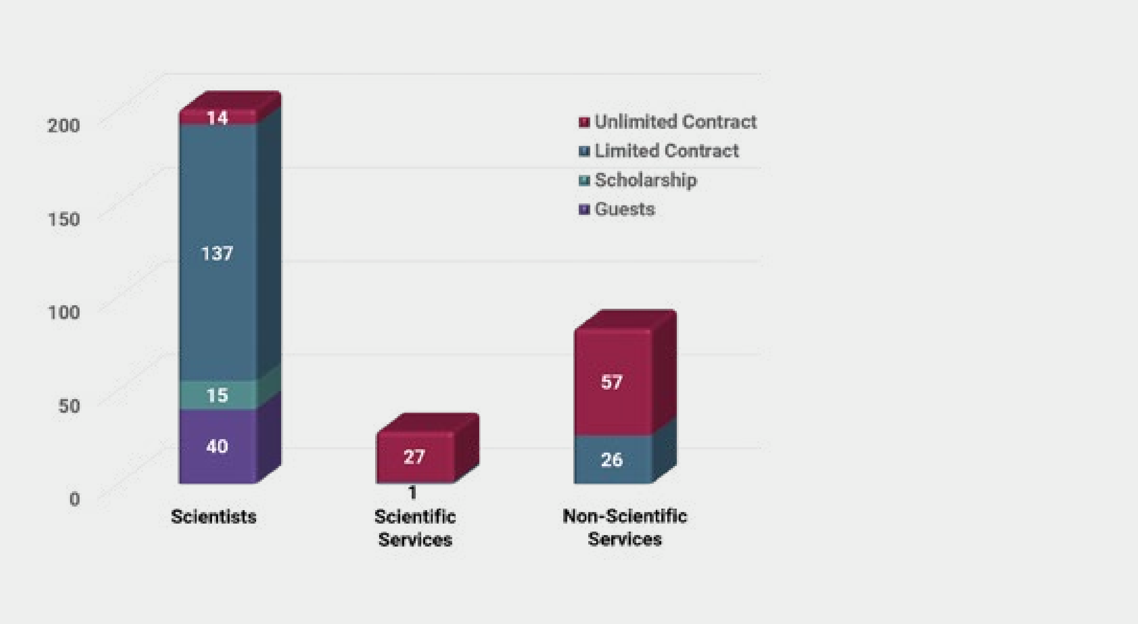
NRW: State of North Rhine-Westphalia

Others: Diverse expenditure on material costs
(e.g. Humboldt Foundation, Merkle Foundation)

Personnel structure

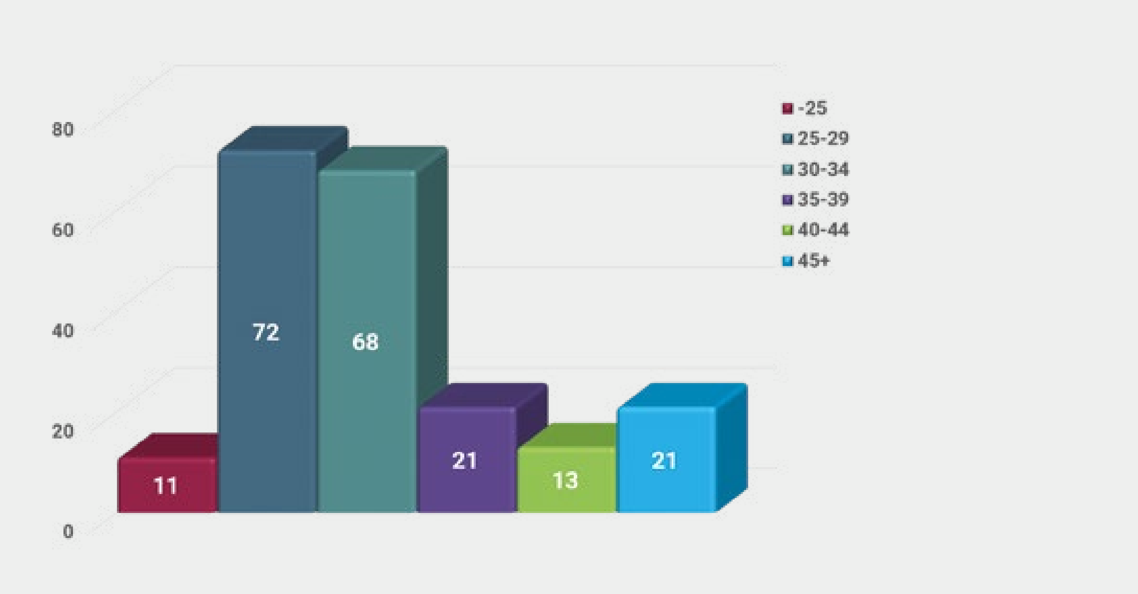
Number of occupied scientific / non-scientific positions

(Absolute numbers, September 2021)



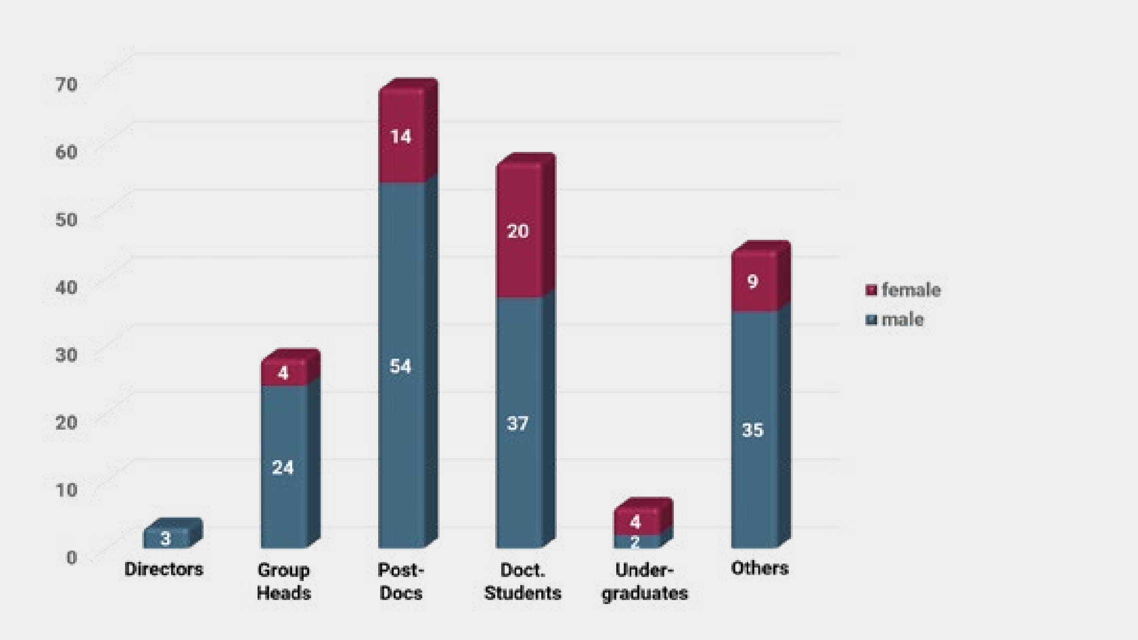
Age distribution of junior scientists

(Absolute numbers, September 2021)



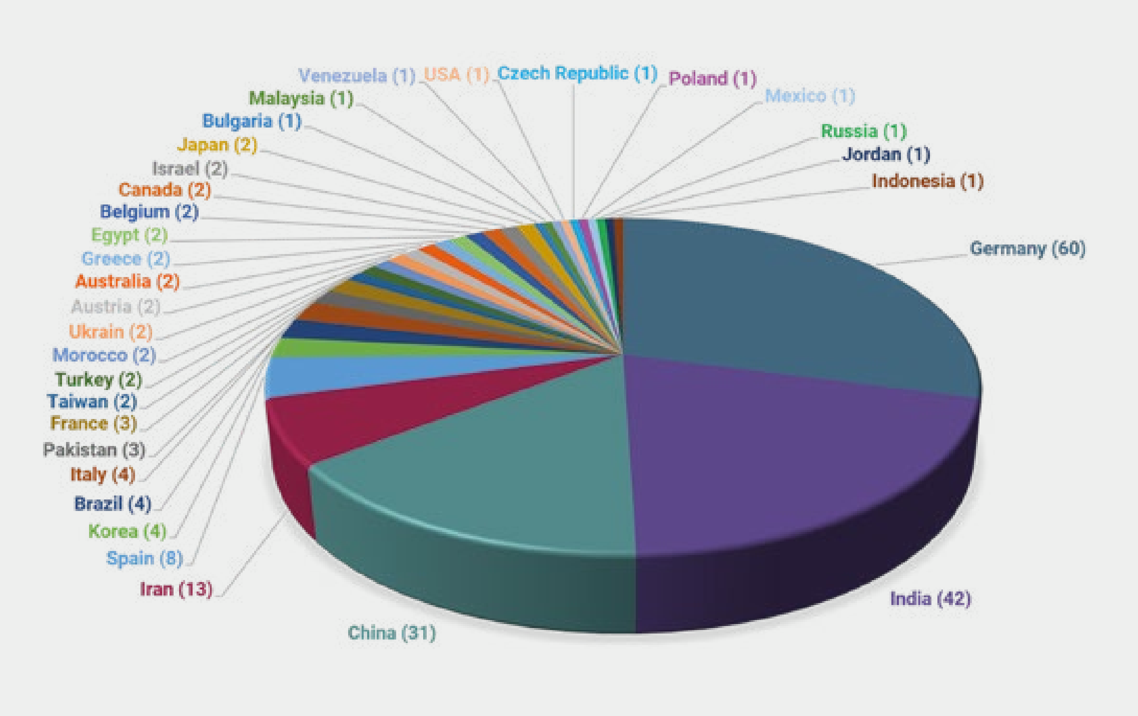
Gender distribution of scientists

(Absolute numbers, September 2021)



Scientists and their home countries

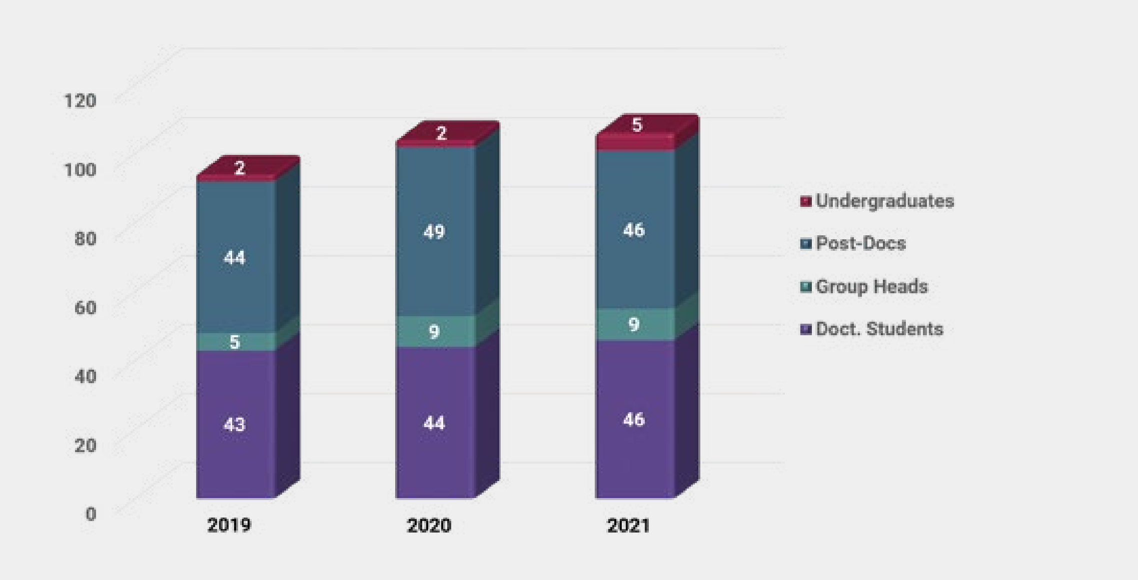
(Absolute numbers, September 2021)



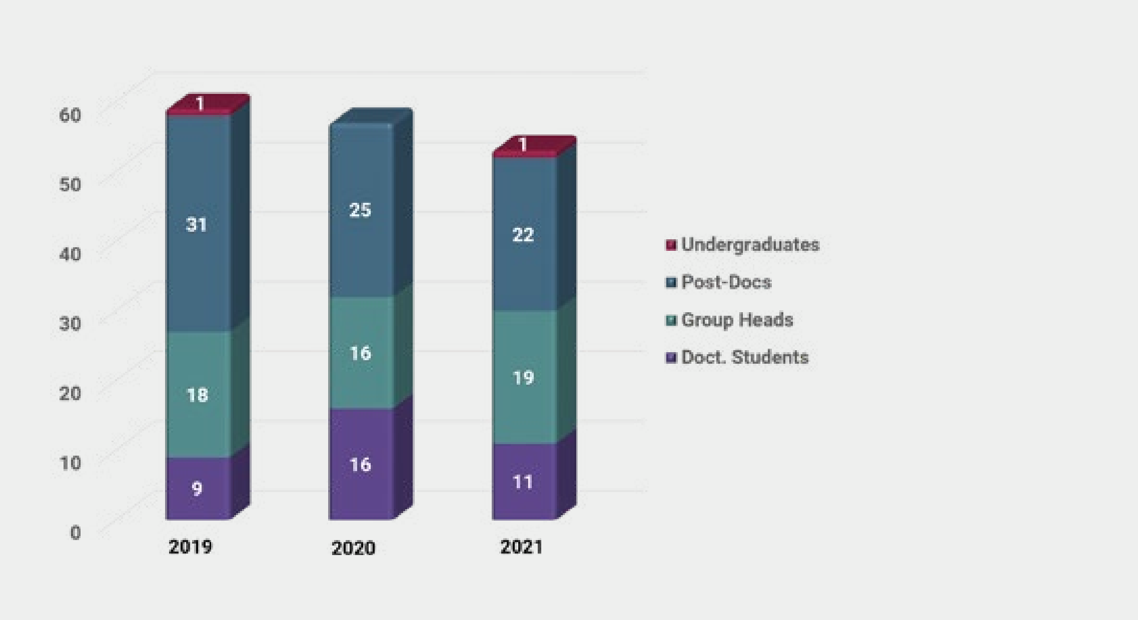
Number of junior scientists

(Absolute numbers, year 2021 data estimated)

Financed via third-party funds



Not financed via third-party funds



Scientific Report
2019 – 2021

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Access

from Central Railway Station:

by Bus 834 (direction Belsenplatz)
to stop Sohnstraße

by Taxi to Sohnstraße/Max-Planck-Straße,
10 minutes

from Airport:

by Taxi to Sohnstraße/Max-Planck-Straße,
15 minutes