Part I.

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New scientific laboratories and facilities

Long-term oriented method development

Large-scale and networking projects

Work, family and campus life

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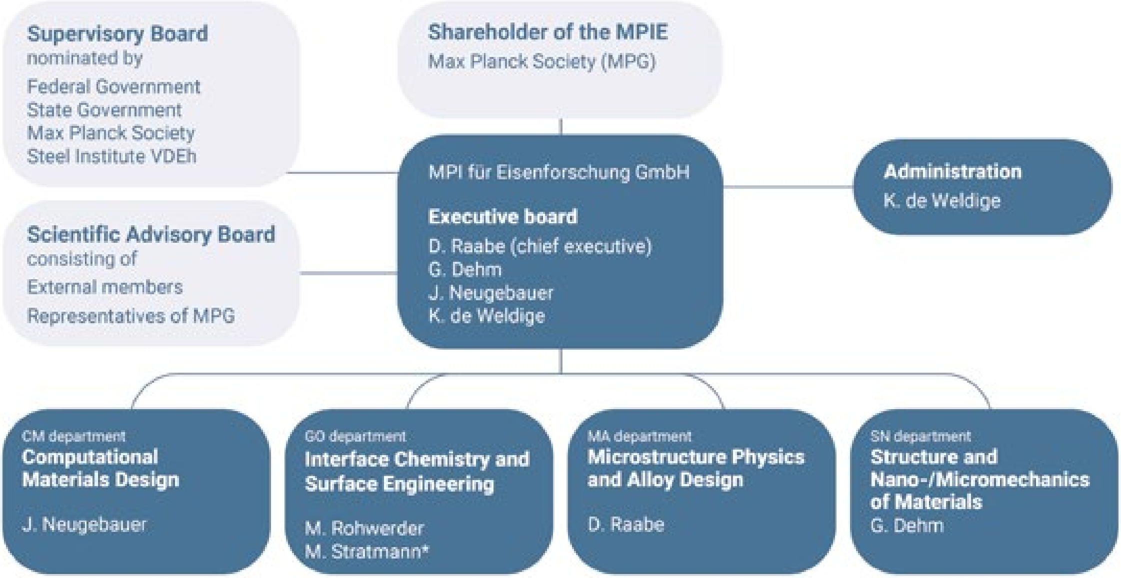
Management of the institute

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.

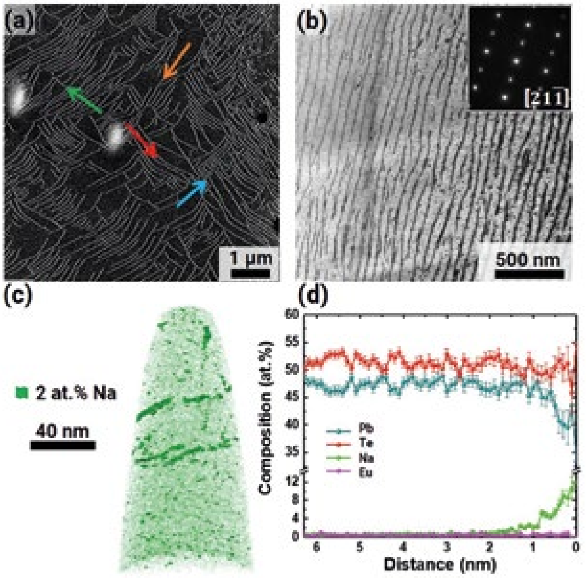


Fig. 2: *a) ECCI image showing four groups of dislocations along <111> directions. b) Bright field TEM image revealing a high density of dislocations (inset: selected area diffraction pattern along the* $[21\bar{1}]$ *zone axis). c) APT reconstruction demonstrating Na enrichment at the dislocations.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

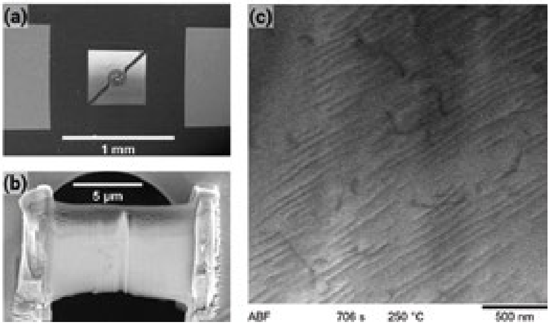


Fig. 3: *a) In situ heating chip with b) FIB-prepared sample. c) An excerpt from in situ heating video showing dislocation networks in an* $Eu_{0.03}Na_{0.025}Pb_{0.945}Te$ *alloy which are immobile during 250 °C isothermal annealing. Data from [32].*

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.

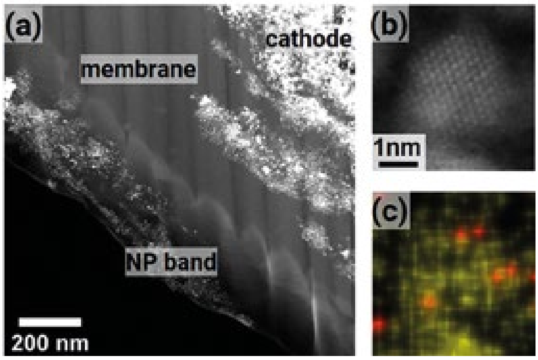


Fig. 4: *a) High-angle annular dark-field (HAADF) STEM micrograph showing a metallic band next to cathode-membrane interface after accelerated stress test. b) STEM and c) EDX of a selected NP within the band. Red colour represents Ru, yellow colour Pt.*

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].

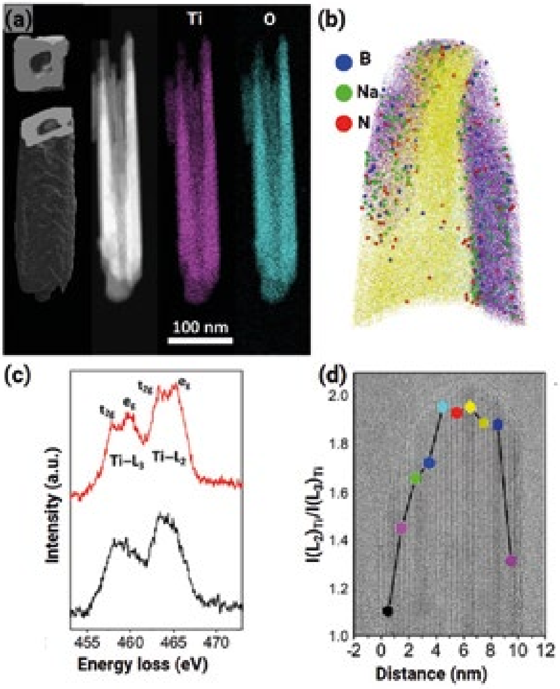


Fig. 5: *a) Electron tomographic reconstruction and EELS elemental maps of a hollow, black TiO_2 nanowire. b) APT reconstruction showing the enrichment of B, Na and N at the TiO_2 surface. c) ELNES of Ti-$L_{2,3}$ edge revealing the presences of Ti^{4+} and Ti^{3+} ions. d)HRTEM image superimposed with 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].

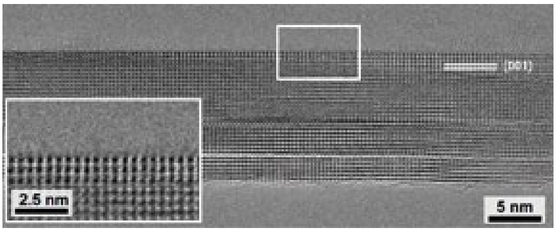


Fig. 6: *HRTEM micrograph of a Nb_3O_7OH nanowire showing the presence of planar defects and the surface structure (magnified shown as inset).*

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

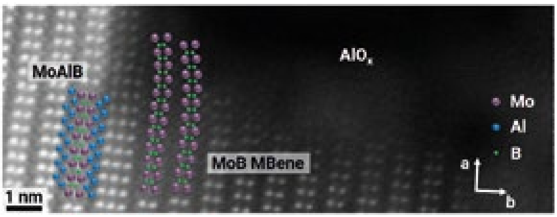


Fig. 7: *Aberration-corrected STEM image revealing the local formation of MoB MBene layers in MoAlB adjacent to amorphous AlO_x region. Data taken from [50].*

(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).

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Max Planck fellow group: Self-reporting materials

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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.

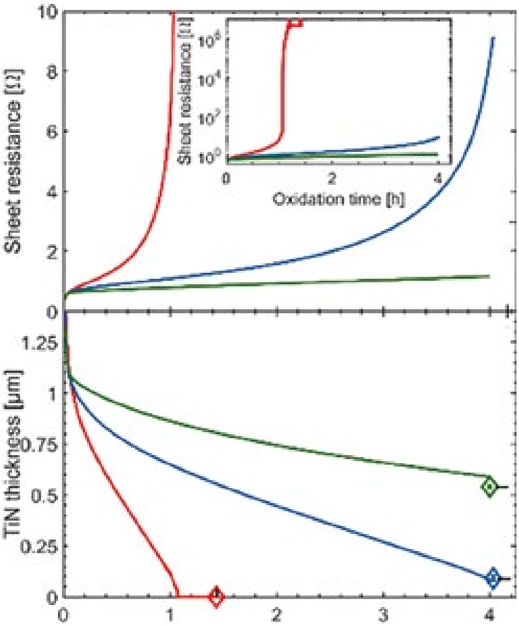

Fig. 1: *Top: in situ measured sheet resistance of TiN thin films over oxidation time for three samples oxidized at 600, 650, and 700 °C; Bottom: remaining TiN thicknesses depending on oxidation time calculated from measured sheet resistances. Diamonds indicate remaining TiN film thickness measured ex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

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Group head: K. Tschulik

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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.

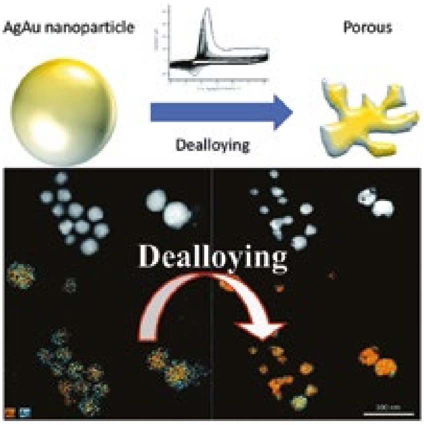

Fig. 1: *Schematic representation of nanoparticle modification by dealloying, cyclic voltammetry data and identical location STEM images and EDX mapping, before and after dealloying of AgAu nanoparticles [1].*

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].

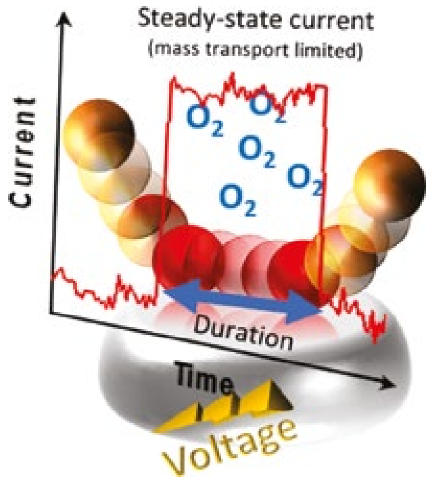

Fig. 2: *Scheme of single particle electrocatalysis at individual NPs.*

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.

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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].

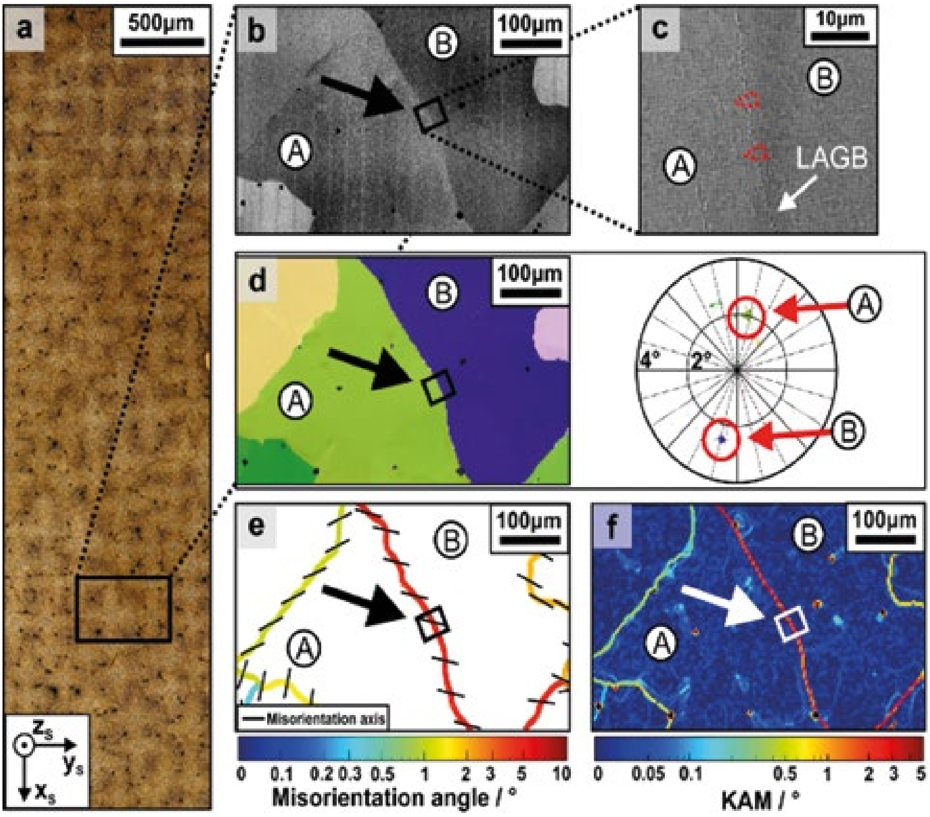

Fig. 1: *Identifying locations for subsequent APT analysis [1]. (a) Optical micrograph of dendritic microstructure. (b) Backscatter electron) (BSE) overview image. (c) Higher magnification micrographs showing a low angle grain boundary between the regions marked with A and B. APT specimens containing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

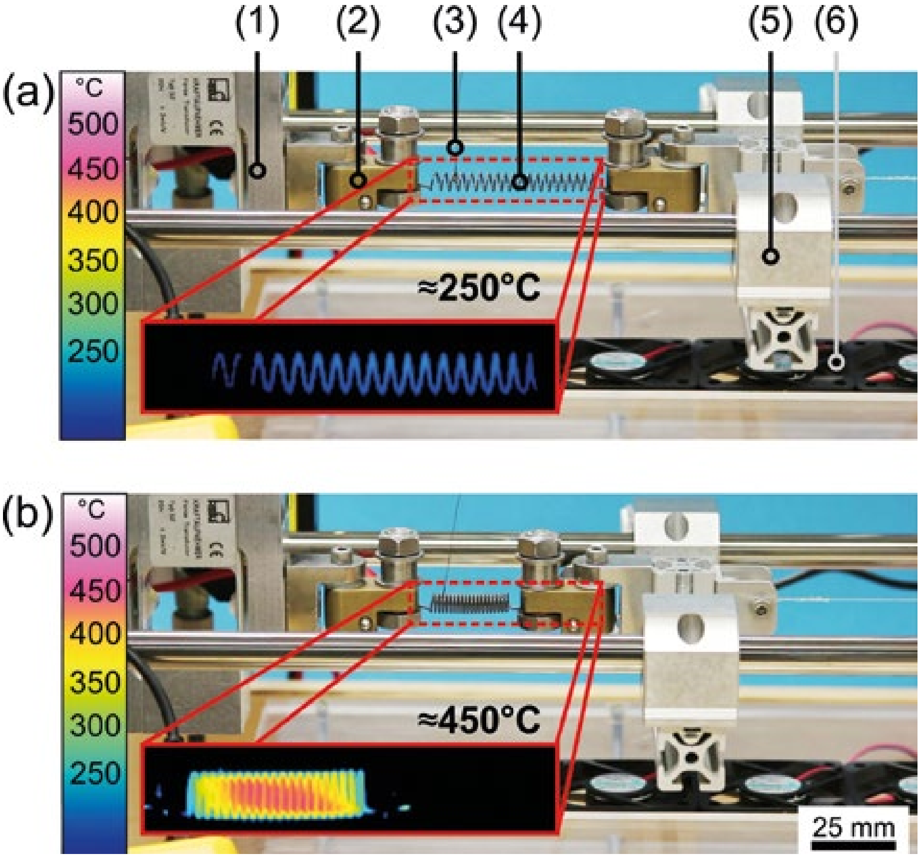

Fig. 2: *Test rig for assessing Ti-Ta HTSMA spring actuator performance [6]. The two photographs were taken during a heating cycle. The insets show colour coded temperature distributions recorded with an IR thermo camera. Key elements of the test rig are highlighted in a) 1 – load cell; 2 – grips (for 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.

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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.

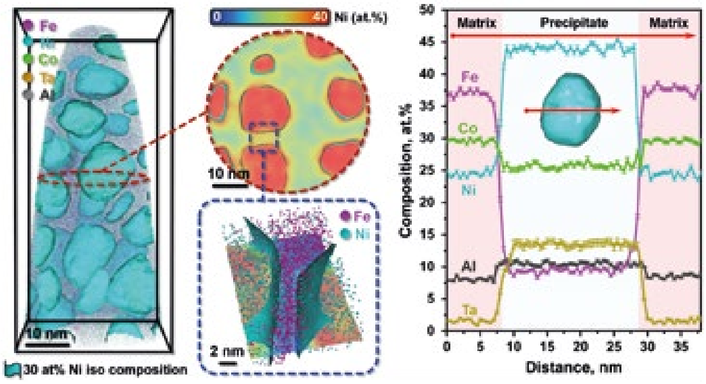

Fig. 1: *Ultrastrong and ductile bulk high-entropy alloys with excellent soft magnetic properties were achieved by introducing a high volume fraction of coherent and ordered nanoprecipitates into the alloy matrix. The high lattice coherency between the homogeneously dispersed nanoprecipitates and the 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.

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MPG group: Thin film materials design

Group head: J. M. Schneider

Materials Chemistry, RWTH Aachen University

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).

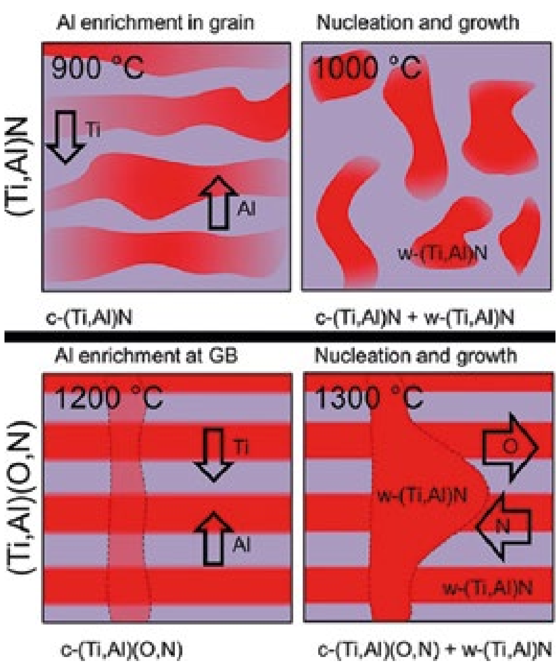

Fig. 1: *Schematic representation of decomposition of rotated c-(Ti,Al)N and c-(Ti,Al)(O,N) coatings; adapted from [1].*

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.

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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].

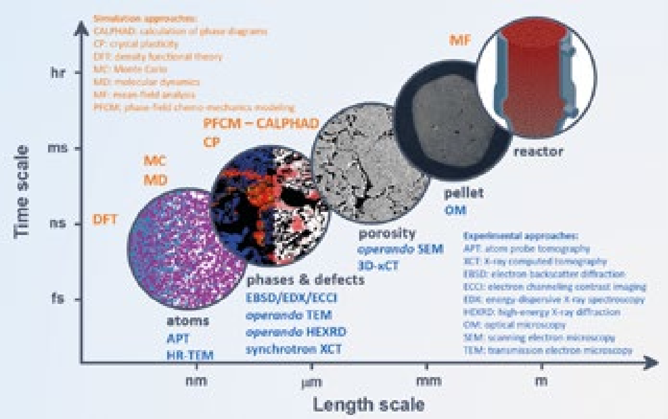

Fig. 2: *Multiscale approaches towards better understanding of the materials' synthesis processes.*

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.

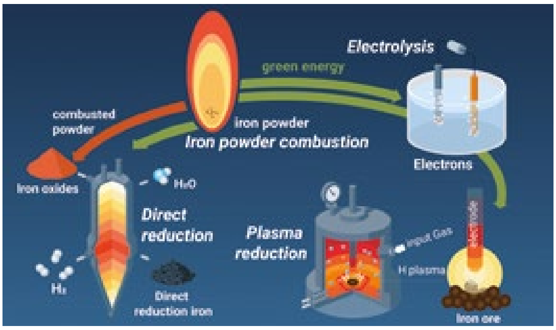

Fig. 1: *The group focuses on the chemical and physical foundations for sustainable synthesis of iron and green energy generation.*

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.

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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.

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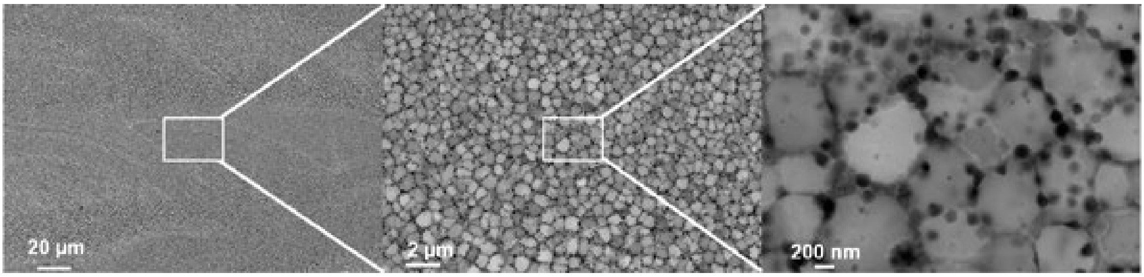

Fig. 1: *Novel materials through novel synthesis: the in situ nanostructuring of TiB_2 particles in a ferritic matrix during SLM of an FeTiB alloys ensures a unique property combination of high stiffness and strength coupled with low density [1].*

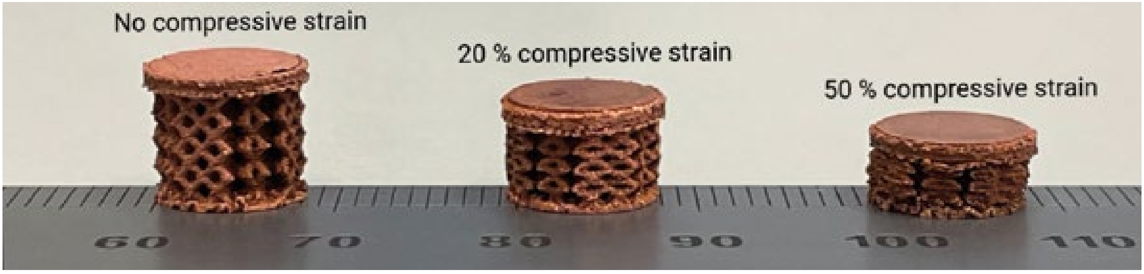

Fig. 2: *Millimeter scale pure Cu octet lattices in the undeformed and after quasi-static deformation at different strains.*

Interdepartmental group: Sustainable material science and technology

Group head: H. Springer

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

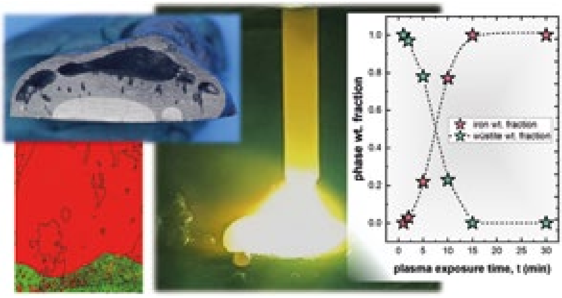

Fig. 1: *Investigating the hydrogen plasma smelting reduction of iron ores at the MPIE: From model processes to microstructure characterisation and evaluation of the reduction kinetics.*

The 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.

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.

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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.

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].

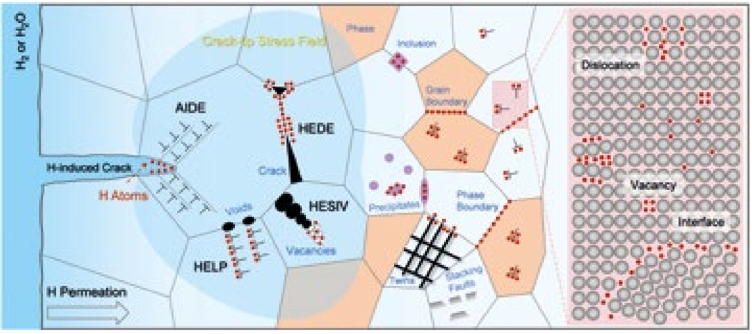

Fig. 1: *Schematic diagram showing the behaviour of H trapping, H-defect interactions and micromechanical models for the formation of H-induced damage in metallic materials.*

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.

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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.

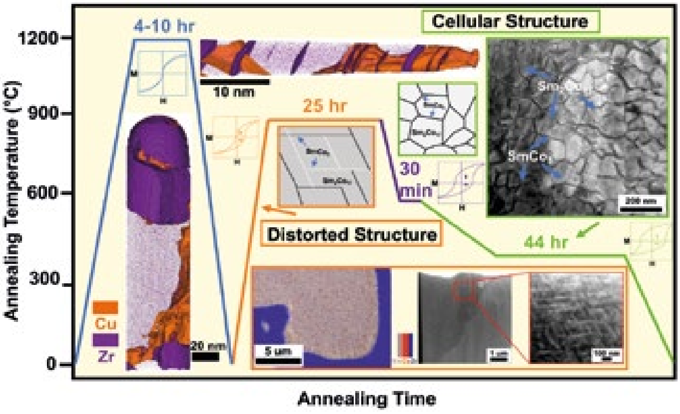

Fig. 1: *Thermomagnetic (Temperature vs time) sequence plot indicating the evolution of nanostructure in a Sm-Co based high-performance permanent magnet. The inset shows elemental distribution maps with Zr and Cu delineated using iso-concentration surfaces highlight the difference between well-formed 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.

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Max Planck partner group: Designing damage tolerant functional oxide nanostructures

Group head: B. N. Jaya

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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.

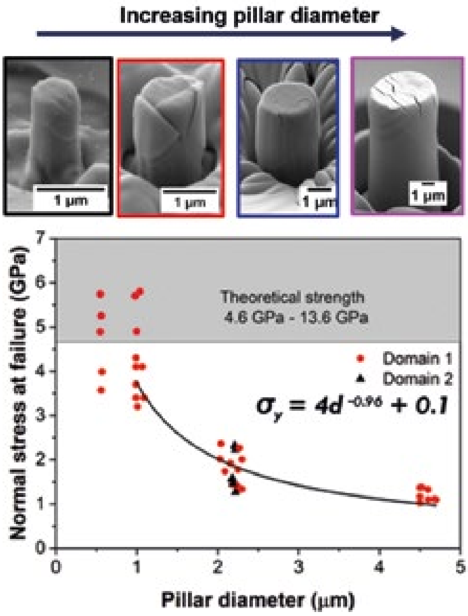

Fig. 1: *Strength and strain tolerance displayed by single crystal micropillars of $BaTiO_3$ as a function of specimen size.*

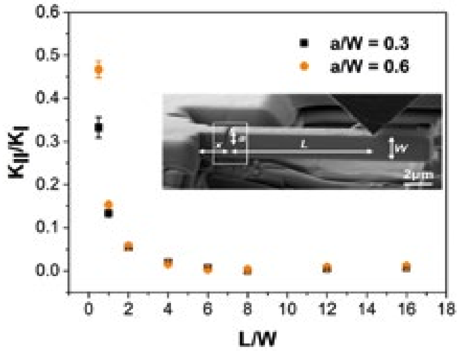

Fig. 2: *Microcantilever fracture test geometry and simulated mode mixity ratios as a function of beam aspect ratio and crack location.*

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.

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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 su- peralloys 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.

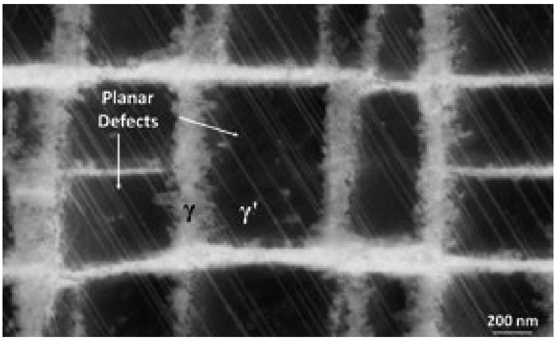

Fig. 1: *An ECCI image of CMSX-4 single crystal Ni-base superalloy after creep deformation at 800 °C under a stress of 800 MPa. It shows bright contrast regions corresponding to planar defects inside cuboidal γ′ precipitates.*

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].

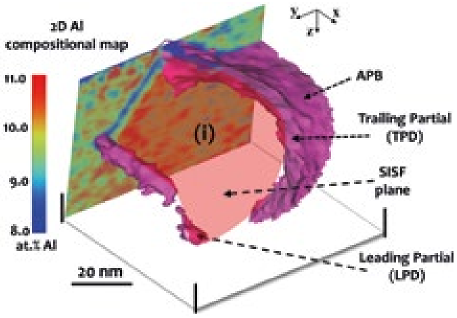

Fig. 2: *A 3D compositional map of planar defects comprising a stacking fault and an anti-phase boundary with their partial dislocations [5].*

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.

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

Group head: S. R. Meka

Indian Institute of Technology Roorkee, India

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].

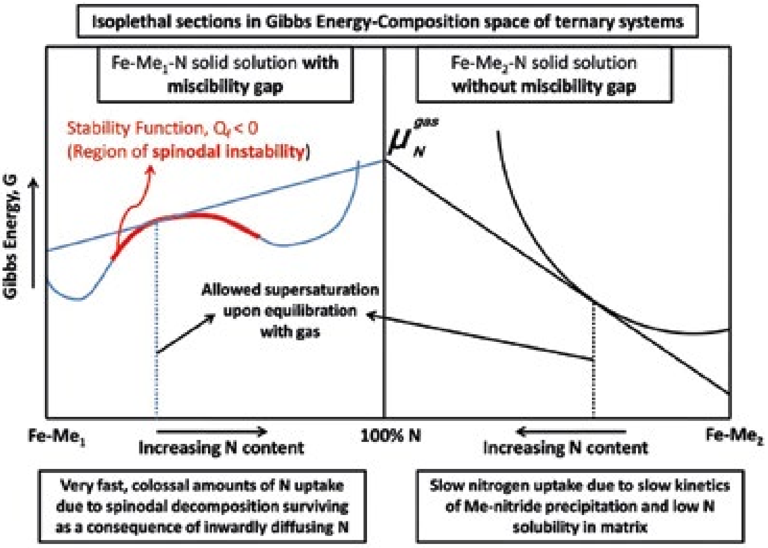

Fig. 1: *Schematic representation of the fundamental difference in thermodynamic properties of alloys exhibiting rapid and weak nitridation kinetics Me$_1$ represents alloying elements Al, Si and Mo and Me$_2$ represents alloying elements Cr, V and Ti. represents the chemical potential of N in the gas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

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.

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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.

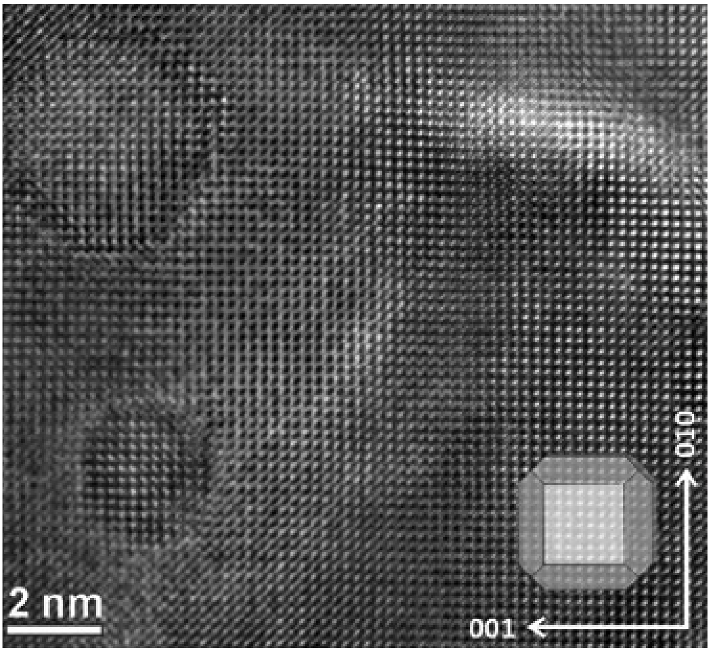

Fig. 1: *A transmission electron microscopy image of a W single-crystal with facetted He voids, interrupting the W crystal lattice. The voids are a combination of a cube {100} and rhombic dodecahedron {110}.*

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.

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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.

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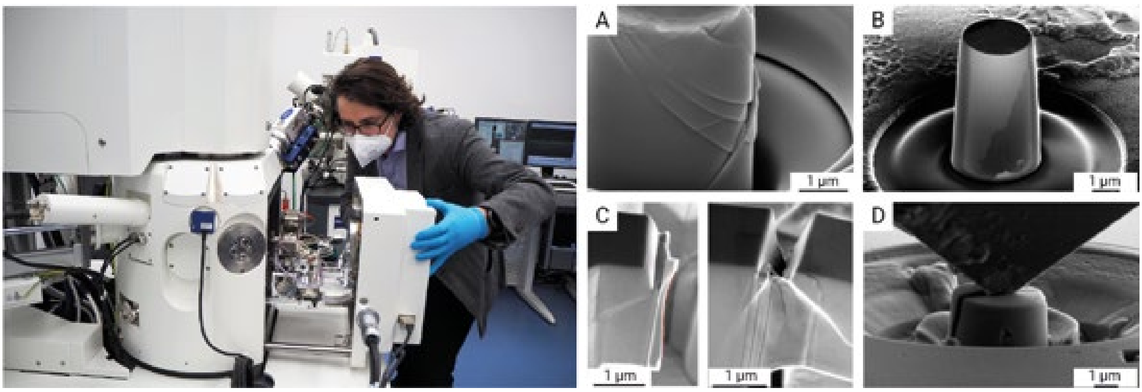

Fig. 1: *Combining broad materials science questions with advanced in situ nano- and micromechanical testing capabilities. Examples from the group's research include deformation in metallic glasses (A); copper bi-crystal milled from a thin-film containing an incoherent twin boundary (B); grain boundary 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.

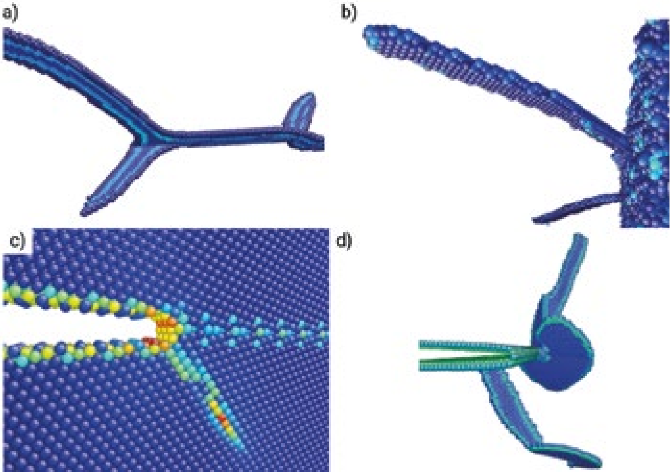

Fig. 1: *Examples of atomistic simulations of different defect-defect interactions: a) dislocation-dislocation interaction, b) dislocation-grain boundary interaction, c) grain boundary fracture and twin emission, d) dislocation – crack interaction and stimulated nucleation of dislocations.*

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.

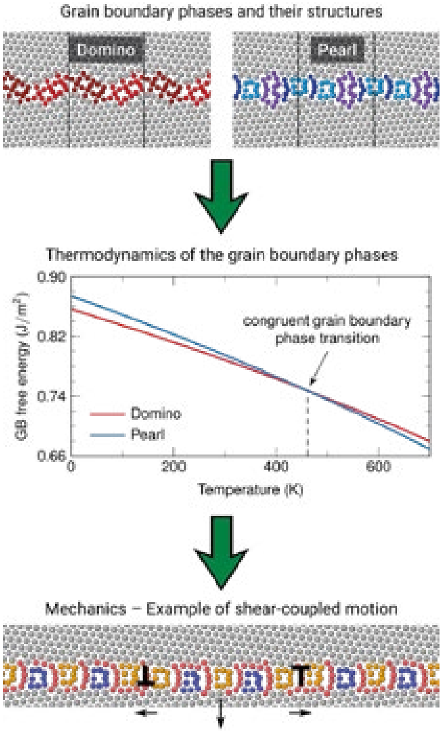

Fig. 1: *Example of the approach towards structure–property relations for GBs. First, the atomic structures of possible GB phases have to be modelled, followed by free energy calculations to understand phase stability and transitions. Finally, mechanical properties, such as the GB mobility, can be investigated 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 $\Sigma37c$ ⟨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).

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Integrated computational materials engineering

Group head: M. Diehl

MA, now at KU Leuven (Belgium)

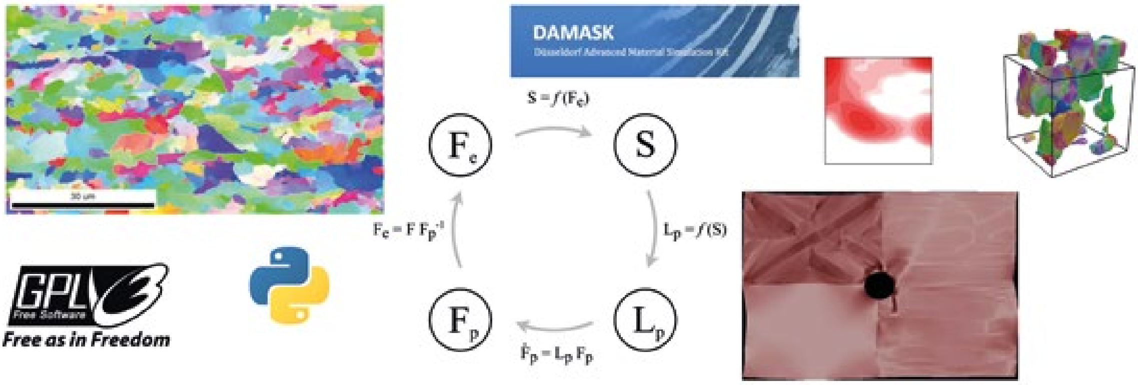

Fig. 1: *The research focus of the ICME group: Development of constitutive laws and numerical methods and integrating them into the free and open source package DAMASK to solve questions in materials science and engineering.*

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.

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Hydrogen mechanics and interfaces

Group head: M.J. Duarte Correa

SN

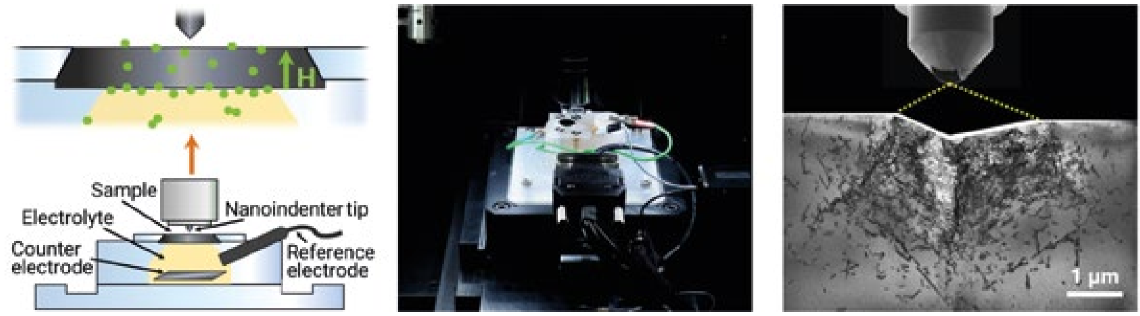

Fig. 1: *Novel back-side hydrogen charging setup developed in-house for in situ micromechanics during hydrogen charging and transmission electron microscopy image of a deformed microstructure after nanoindentation under the influence of hydrogen.*

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).

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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.

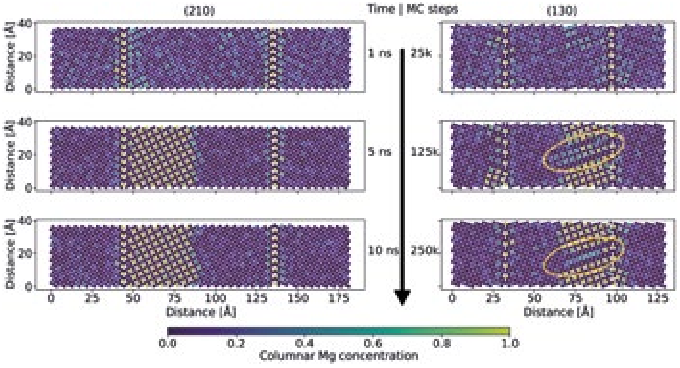

Fig. 1: *Coupled molecular dynamics/Monte Carlo simulations showing how grain boundary geometry in Al can promote the formation of a pristine Mg-rich phase, or impose persistent defects (circled yellow) in the secondary-phase. Al shown dark, Mg shown light, with columnar chemistry averaged along the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

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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.

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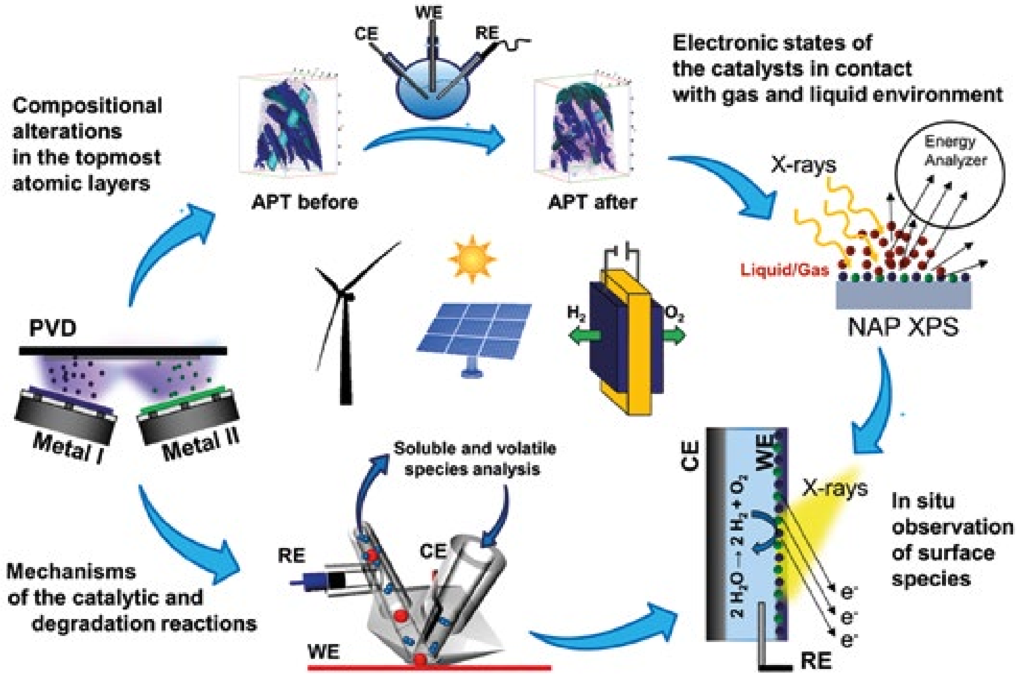

Fig. 1: *Schematic presentation of the approach to achieve the goals of the group 'Dynamic Electrocatalytic Interfaces.'*

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.

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 thermomechanical 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].

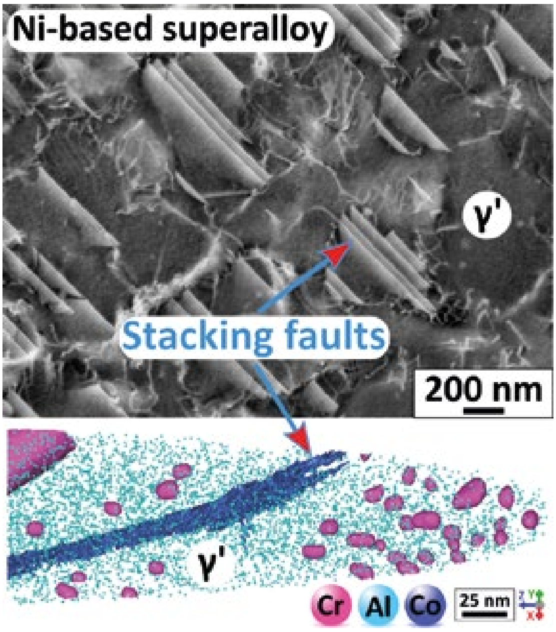

Fig. 1: *Electron channelling contrast image of a deformed superalloy after creep at 750 °C showing stacking faults in γ' precipitates, alongside a 3D-atom probe reconstruction revealing segregation of cobalt at stacking faults.*

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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.

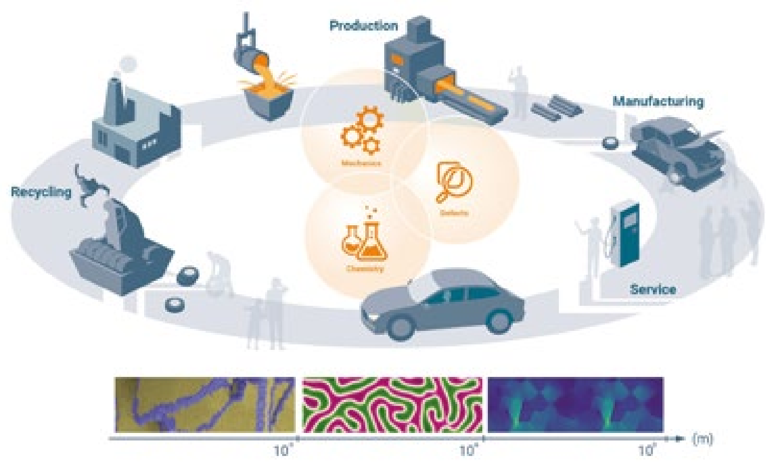

Fig. 1: *Schematic of metals’ life-cycle. Investigating this cycle requires understanding of chemistry, mechanics and microstructure. The theoretical and computational study of new low-carbon footprint alternatives in metal production and recycling is the focus of this group.*

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.

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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].

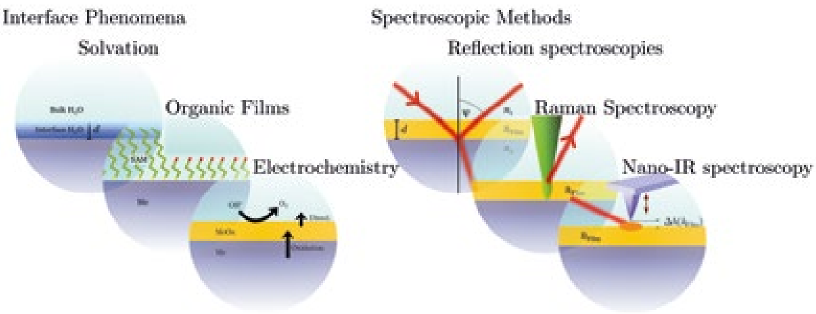

Fig. 1: *Studied interface phenomena and methods employed in the group.*

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.

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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].

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].

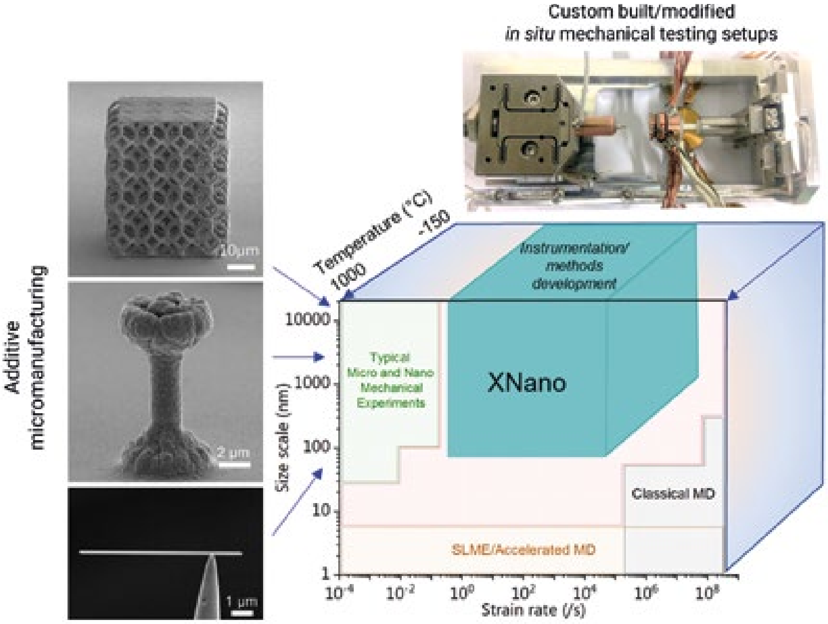

Fig. 1: *Summary figure showing the core foci of the XNano group in pushing the boundaries of small-scale mechanics towards higher strain rates (and high/cryo temperatures) and small-scale manufacturing towards complex 3D architectures.*

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.

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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).

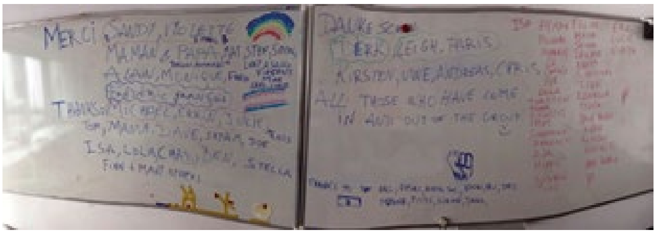

Fig. 1: *White board in my office that I used as background during the online ceremony in November 2020, thanking (almost) all those who helped and contributed to my adventures.*

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].

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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.

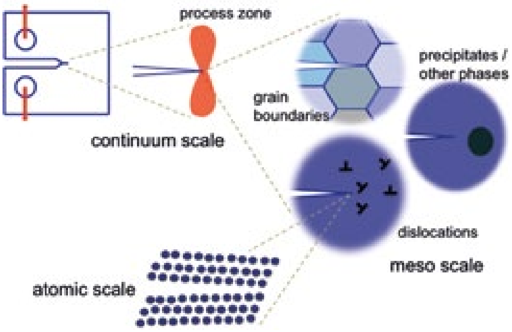

Fig. 1: *Relevant scales and microstructural features that determine the response of a crack to an external load.*

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 micro-K_{Ic} and now allows for the first time fully 3D studies of fracture and crack-tip plasticity at the mesoscale.

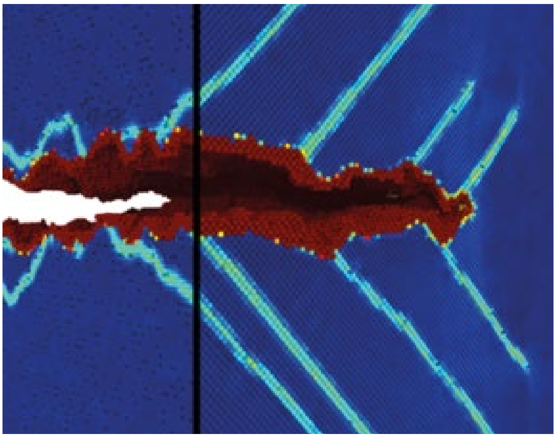

Fig. 2: *Part of a large-scale 3D fracture simulation in off-stoichiometric B_2 NiAl showing how atomic-scale heterogeneities can influence the crack propagation and dislocation emission (atoms coloured by atomic strain).*

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.

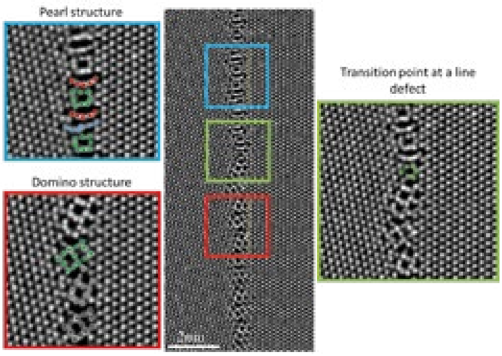

Fig. 1: *Scanning transmission electron microscope image of an asymmetric ∑19b [111] tilt GB in Cu consisting of pearl and domino structure separated by a disconnection.*

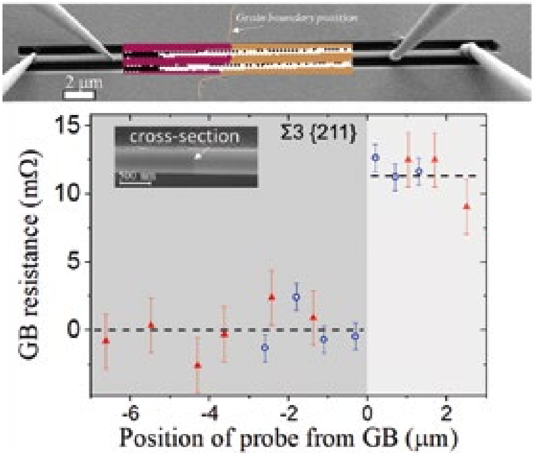

Fig. 2: *Four point in situ resistance measurement of a GB. Electron microscopy and diffraction is used to determine the GB character and position. A clear resistance signal is obtained for the incoherent ∑3 [111] $(\bar{2}11)$ GBs (red and blue data points).*

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).

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ERC-Consolidator Grant: Seeing hydrogen in metals

B. Gault

MA

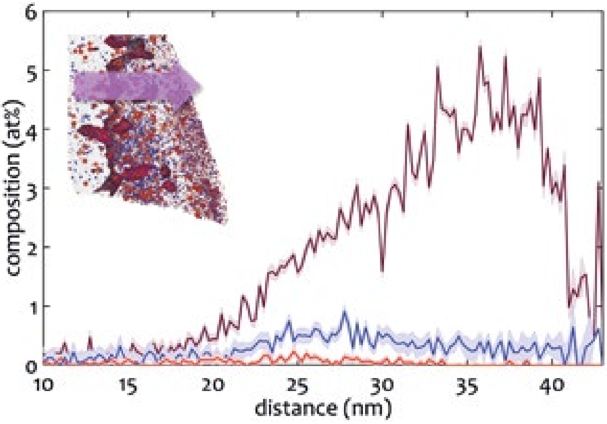

Fig. 1: *Distribution of D and H measured by APT at the interface between ferrite and decomposed cementite. [Ref. 3].*

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.

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).

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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.

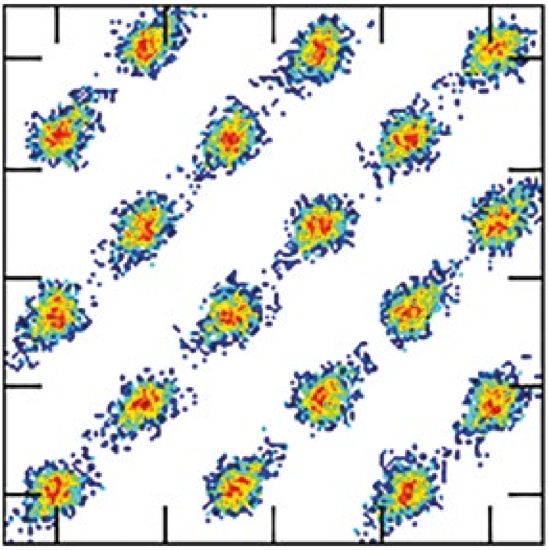

Fig. 1: *Two-dimensional histogram of ab initio molecular dynamics trajectories of a simulation for bcc Ti at 1025 K. The trajectories reveal a fingerprint of the martensitic transformation to the low-temperature hexagonal omega phase. Within TIME-BRIDGE new insights about this transformation have been 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).

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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: $<1\bar{1}00>$ and $<11\bar{2}0>$. 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.

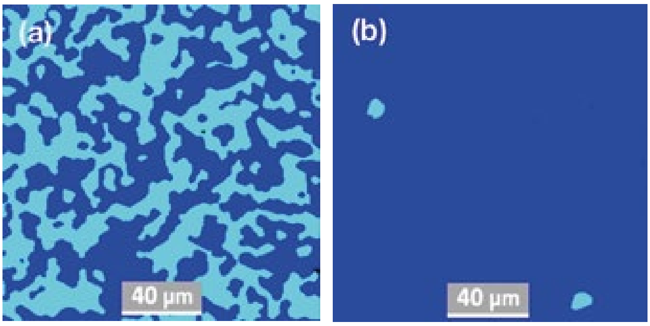

Fig. 1: *Preliminary results with Cu on sample with the two different miscut c-sapphires; EBSD maps of (a) a Cu maze <111> bi-crystal film on c-sapphire; (b) single-crystal with some residual "islands" of the second orientation. The two different blue colours correspond to the two twin-related grain 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 $<1\bar{1}00>$ directions at sapphire surfaces. However, if the miscut reveals $<11\bar{2}0>$ 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.

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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.

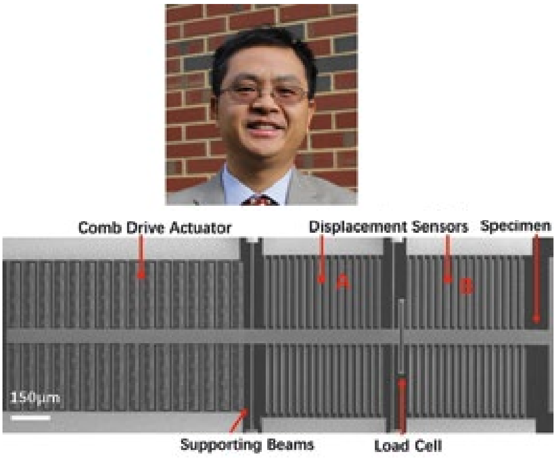

Fig. 1: *Picture of Prof. Yong Zhu and the MEMS-bases nanomechanical testing apparatus he developed. This system is utilized in the in situ heating and biasing holder of MPIE to probe atomic scale deformation processes in aberration-corrected TEMs.*

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.

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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.

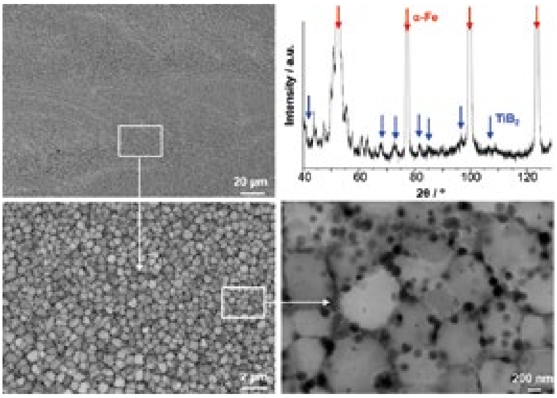

Fig. 2: *Ideal match between novel material concepts and synthesis technologies: in situ nano structuring of high modulus steels during additive manufacturing [3].*

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.

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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.

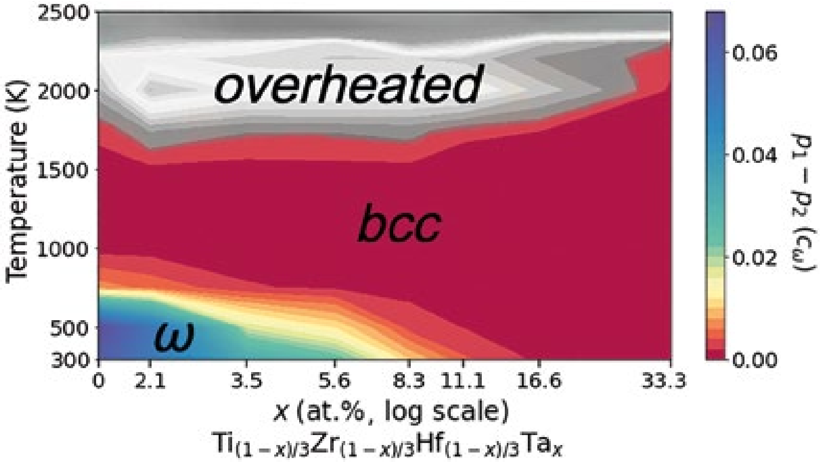

Fig. 1: *Application of developed active learning machine-trained potentials to explore the phase stability of TiZrHfTa$_x$ depending on Ta concentration x and temperature [5]. The different structural phases, i.e., omega and BCC, can be clearly distinguished based on a developed structural descriptor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

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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

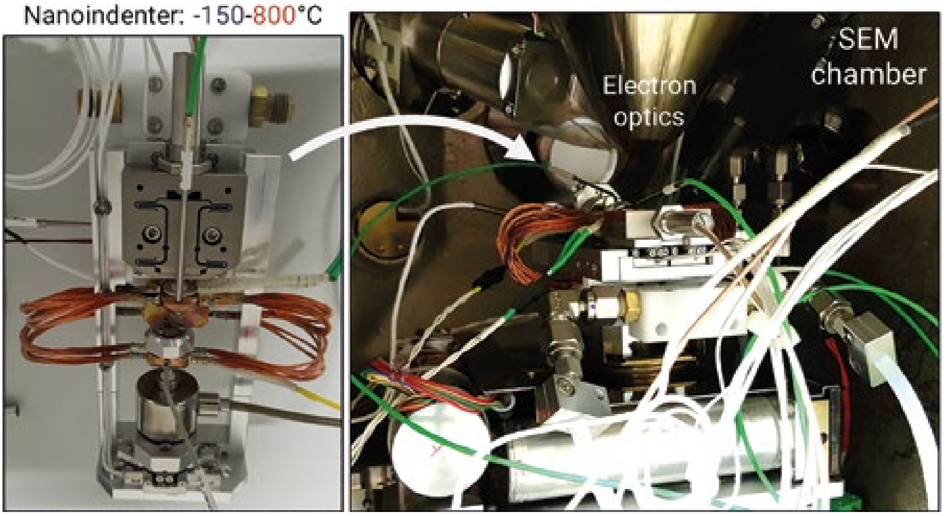

Fig. 1: *The nanoindentation system set-up for cryo temperature and quasi-static indentation of fused quartz within a tungsten-filament SEM. The modular system can be modified for heating, cooling or high-speed measurements and the requisite water-cooling tubes and cabling fed-through the uniquely designed 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

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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.

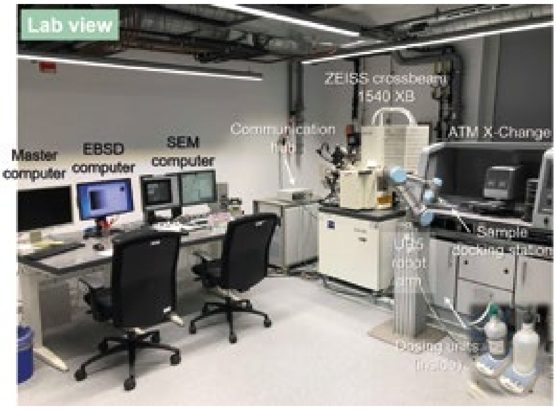

Fig. 1: *Overview on the large-volume 3D EBSD setup based on chemo-mechanical serial sectioning.*

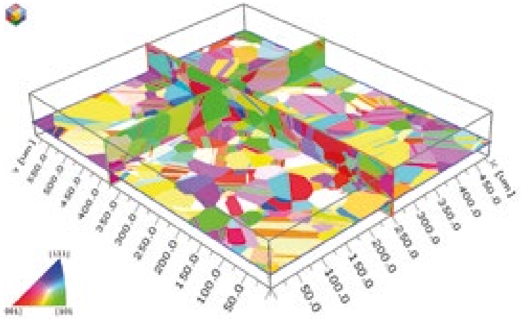

Fig. 2: *3D EBSD data set of a 316 stainless steel, obtained with the large volume 3D EBSD system. The microstructure is displayed and analysed with the program QUBE, programmed by P. Konijnenberg.*

- 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. 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.

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*.

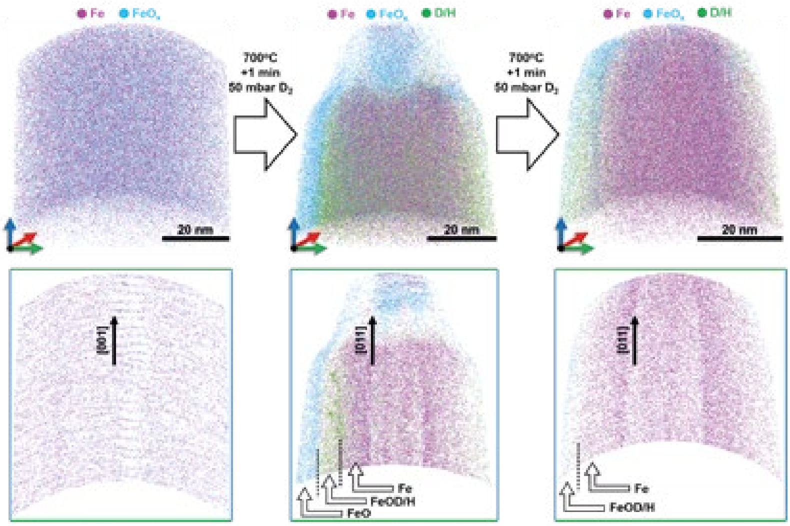

Fig. 1: *Transient state captured during the direct-hydrogen reduction of FeO with the RHM.*

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 precooled 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.

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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.

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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).

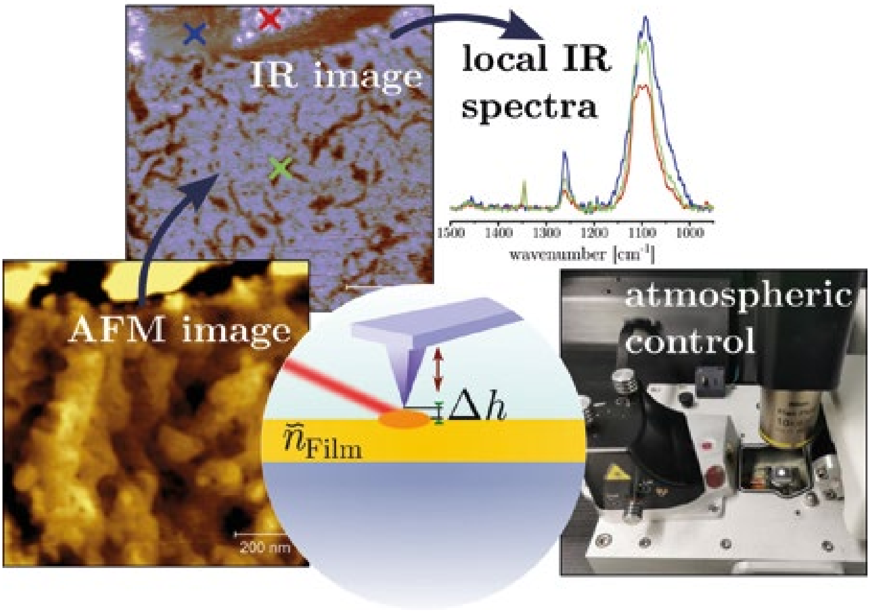

Fig. 1: *Illustration of the measuring principles of photothermal AFM-IR and photo of open sample chamber with piezo stage and measurement head. AFM and spectral data obtained from an organic monolayer of oligo-ethyleneglycol-terminated thiols on gold (111). The IR intensity image measured at 1347 cm^{-1} 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 multiscale 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.

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].

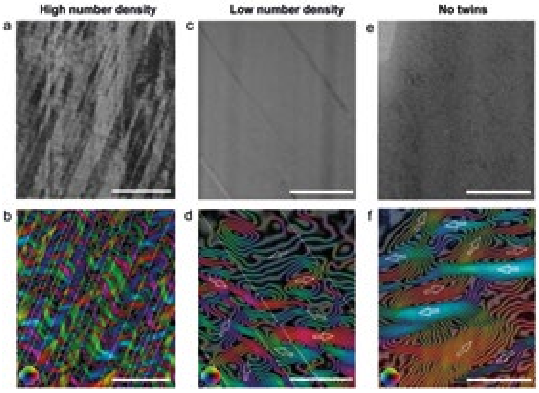

Fig. 1: *Magnetic domain structure of τ-MnAl with high- and with low-densities of micro-twins. (a, c, e) TEM images and (b, d, f) the corresponding in-plane magnetic induction map of magnetic domains extracted from off-axis electron holography.*

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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).

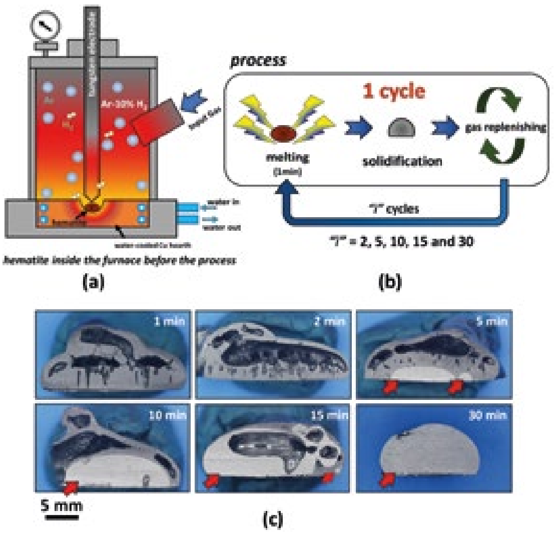

Fig. 1: *Experimental model process for the hydrogen plasma smelting reduction [3]: (a) modified arc-melter, (b) scheme of kinetic experiments, and (c) cross section showing the progressing reduction of liquid iron ore.*

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Challenges of ECCI on light metals

S. Zaefferer, S. Nandy, C. Broß

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.

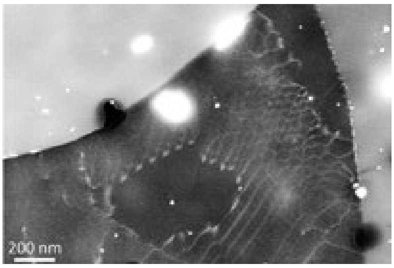

Fig. 1: *ECCI of a small angle grain boundary in a 7075 Al alloy, imaged at 20 kV. The large angle grain boundary at the right is decorated with newly precipitated particles. of a Zn-rich phase.*

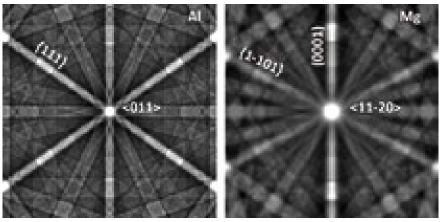

Fig. 2: *ECC patterns simulated with the software EMSoft by Marc de Graef, for fcc Al (atomic number Z=13) and hcp Mg (Z=12). Al shows much stronger, sharper and more homogeneously distributed reflectors.*

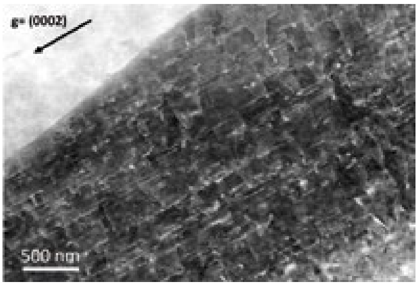

Fig. 3: *ECCI of dislocations in an extension twin of a slightly deformed Mg-Ca-Zn alloy. Imaging with g=(0002) makes <c+a> dislocations visible.*

Additively micromanufactured fluid-filled metal microvessels: APT sample carriers

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

[1]MA, [2]SN

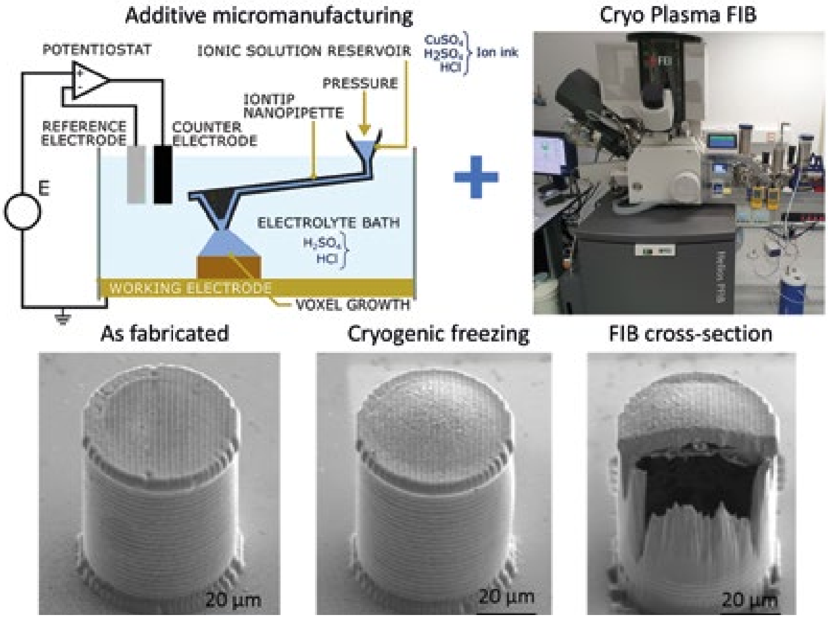

Fig. 1: *Top: Process flow involving additive micromanufacturing followed by cryogenic milling using a PFIB. Bottom: A copper micropillar with encapsulated water, which was subsequently frozen resulting in bulging side and top walls – seen more clearly from FIB cross-section.*

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).

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

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[1]SN, [2]NG

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].

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.

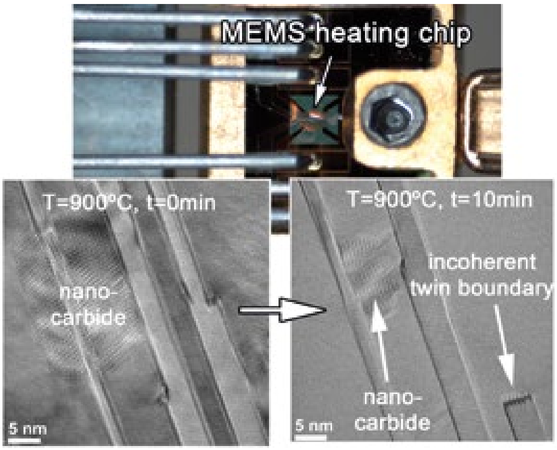

Fig 1: *Top: Picture of the in situ heating holder with electrical contacts and the MEMS-chip, where the FIB-prepared sample is mounted to. Bottom: Two snapshot images of an atomic resolution heating sequence captured at 900°C showing the dissolution of a nano-carbide and associated de-twinning process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

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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.

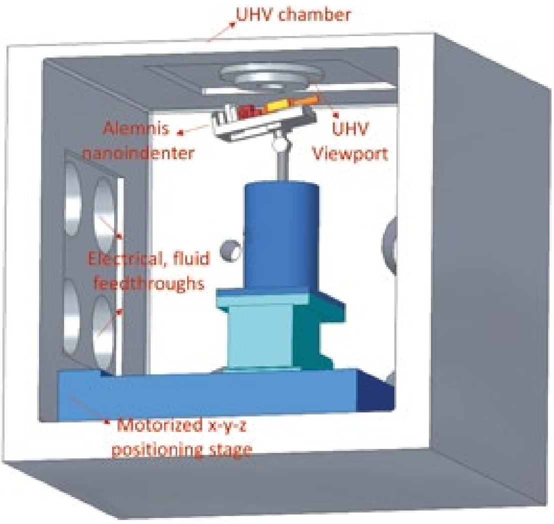

Fig. 1: *CAD design of the environmental chamber for conducting micromechanical experiments under ultra-high vacuum (UHV) or different atmospheres, mimicking real-life working conditions.*

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).

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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*.

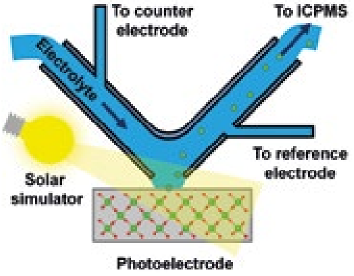

Fig. 1: *Schematic of the illuminated scanning flow cell (iSFC) setup coupled to inductively coupled plasma mass-spectrometry (ICPMS).*

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].

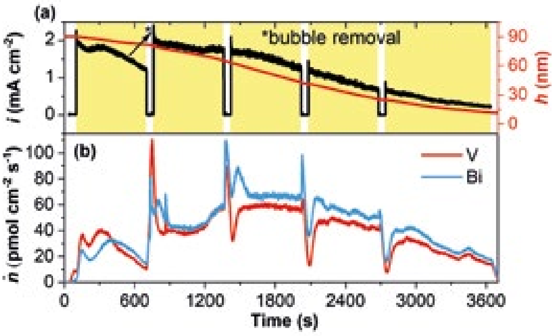

Fig. 2: *A life cycle study on a $BiVO_4$ photoanode [3]. (a) Photocurrent density i (black), film thickness h (red), and (b) dissolution rates of V and Bi from $BiVO_4$ under 1.6 V in borate buffer electrolyte. Windows with 100 mW cm^{-2} illumination are highlighted in yellow.*

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.

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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.

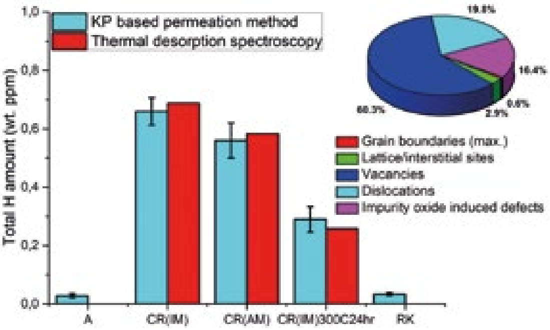

Fig. 1: *A comparison of total hydrogen amount in annealed (A), cold-rolled (CR(IM: induction melted); CR(AM: arc melted)) or re-crystallized (RK) 5 wt.% Ni ferritic steels, obtained by the hydrogen electrode-based method and by thermo-desorption analysis All samples with same thickness (1 mm) were loaded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

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

M. Todorova[1], C. Freysoldt[1], S. Wippermann[2], J. Neugebauer[1]

[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.

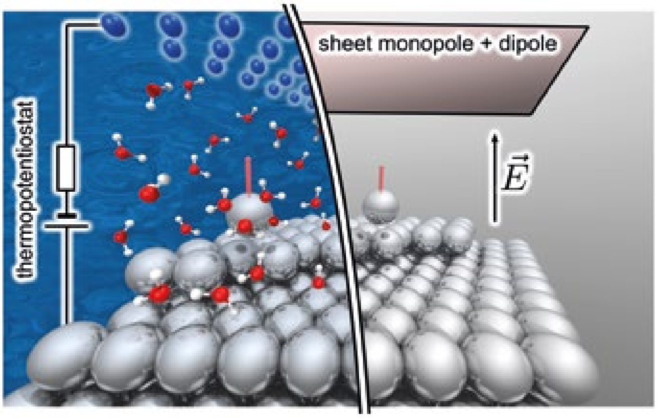

Fig. 1: *Schematic representation of dissolution (in electrochemical environment, left) and field evaporation (right) from a surface under the influence of an applied electric field.*

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.

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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.

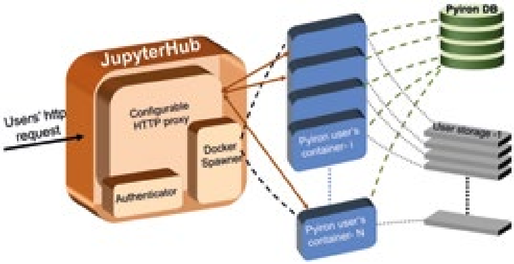
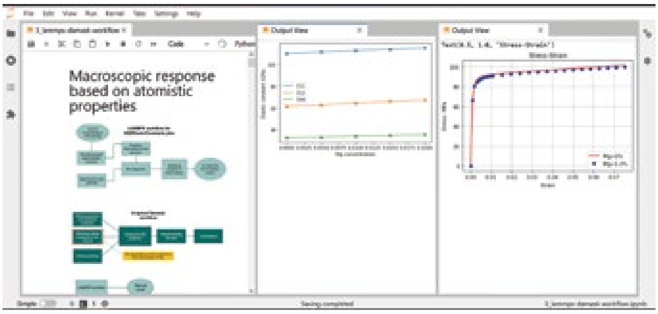


Fig. 1: *The structure of the docker network deployed in the cmserver02. Jupyter docker container authenticates users, and spawns Jupyter containers with preconfigured conda environment.*

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).



Macroscopic response based on atomistic properties

Fig. 2: *Snapshot of Jupyter notebook on the in-house jupyter hub server. The displayed DAMASK simulation determines the elasto-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.

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:

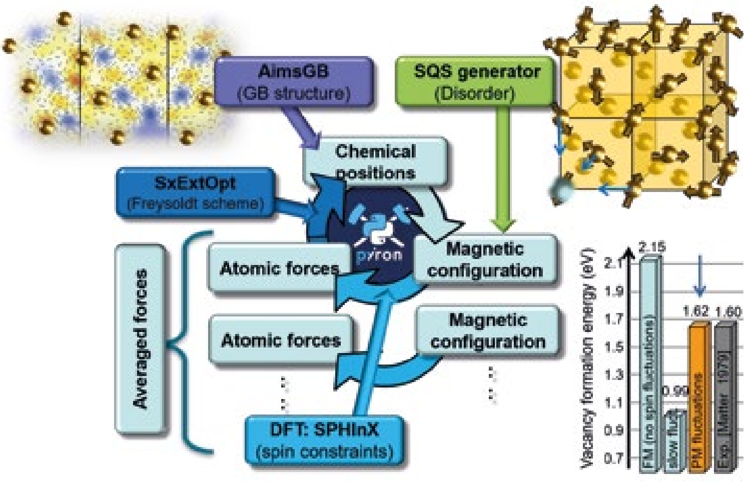


Fig. 1: *Simulation protocol for the atomic relaxation next to defects taking temperature dependent magnetic excitations into account.*

- 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).

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Pyiron – an integrated platform for materials simulations and data management

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

CM

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.

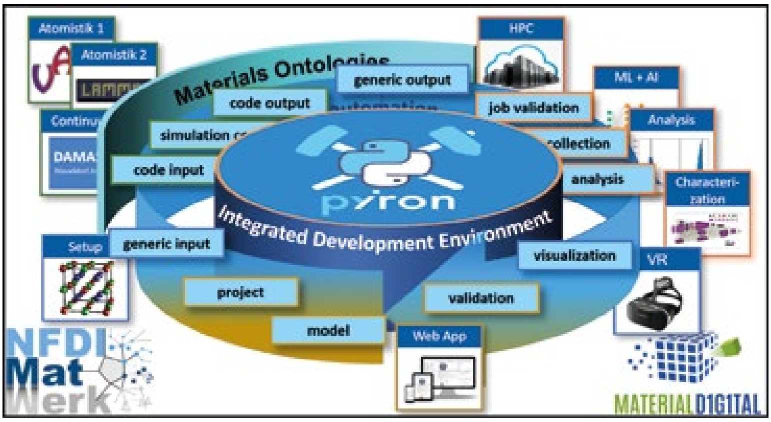

Fig. 1: *Generic workflow for complex simulation protocols in pyiron. Within the pyiron environment a large set of tools becomes accessible, while the technicalities of the interfaces are shielded from the user.*

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.

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The Düsseldorf Advanced Material Simulation Kit: DAMASK

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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.

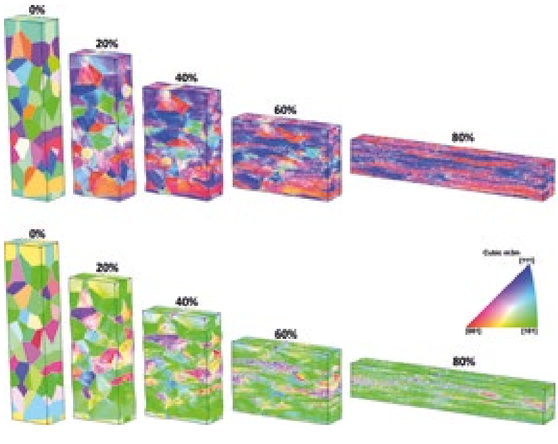

Fig. 1: *DAMASK simulation result for plane strain compression; top row: IPF parallel loading (vertical) direction, bottom row: IPF parallel stretching (horizontal) direction.*

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.

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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.

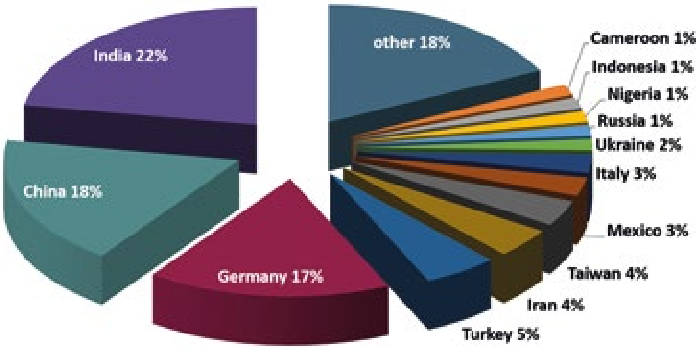

Fig. 1: *Home countries of doctoral students.*

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.

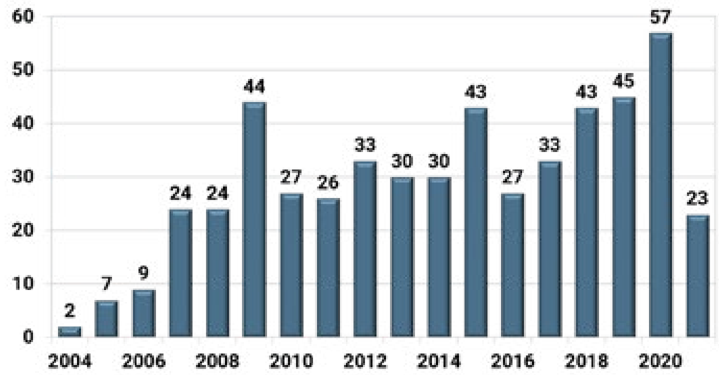

Fig. 2: *Articles in peer reviewed journals.*

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

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.

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:

- 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

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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.

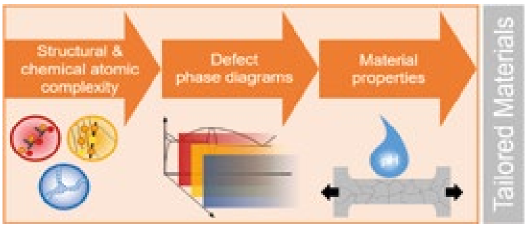

Fig. 1: *Vision of the CRC: Description of the structural and chemical complexity of defects at the atomic scale in defect phase diagrams together with their mechanical and corrosion properties will provide a Materials Design Toolbox enabling materials scientists and engineers to design tailored materials.*

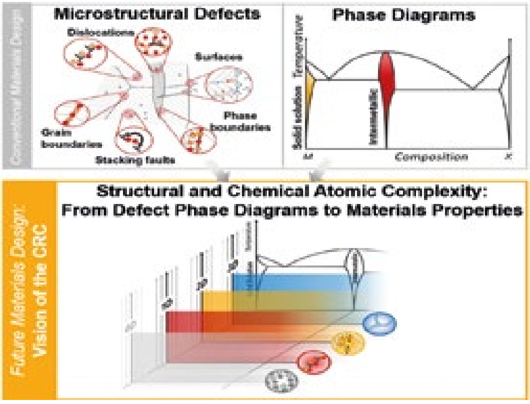

Fig. 2: *Extension of materials design from the currently decoupled approaches considering microstructural defects and phase diagrams towards a comprehensive framework considering both aspects in terms of the full structural and chemical complexity of defects at the atomic scale and their thermodynamic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

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Compositionally complex alloys – high entropy alloys

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N.J. Peter[3], S. Wolff-Goodrich[3], C. Liebscher[3], G. Dehm[3]*

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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.

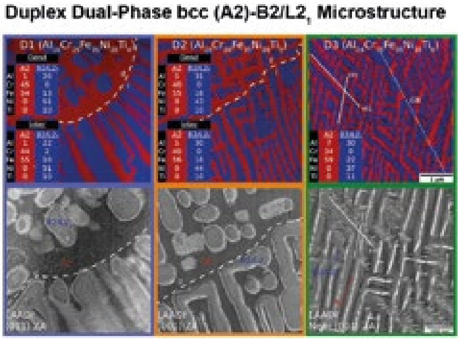
Duplex Dual-Phase bcc (A2)-B2/L2_1 Microstructure

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.

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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.

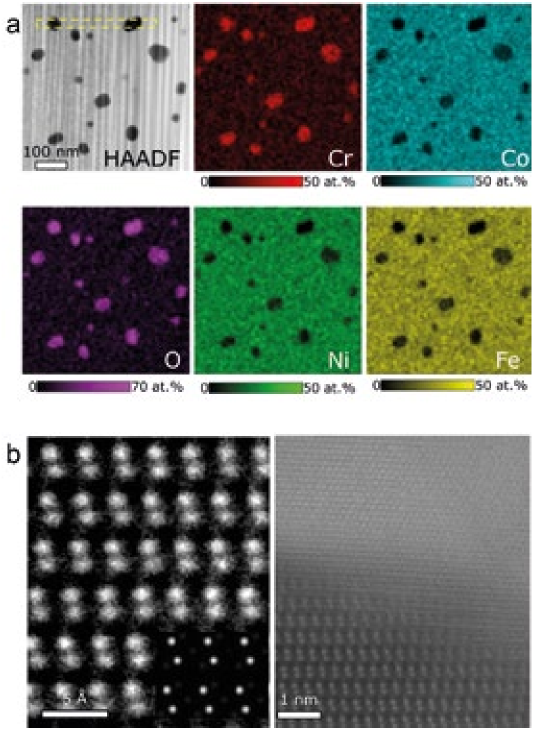

Fig. 1: *(a) Scanning transmission electron microscopy (STEM) Z-contrast image (high angle annular detector: HAADF) showing the distribution of* Cr_2O_3 *nanoparticles and corresponding elemental maps by STEM-energy dispersive X-ray spectroscopy. (b) Atomic resolved STEM-HAADF and annular bright field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

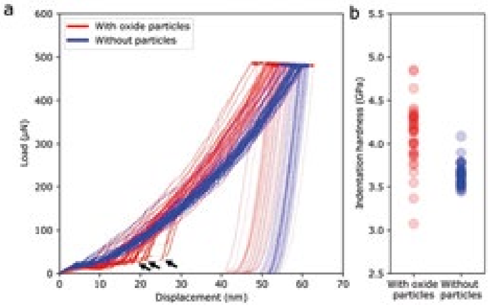

Fig. 2: *(a) Nanoindentation data and b) distribution of nanohardness values show a higher film hardness for the quaternary films with* Cr_2O_3 *particles (red curves and data points) [3].*

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].

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Interdisciplinary Centre for Advanced Materials Simulation

T. Hickel, J. Neugebauer

CM

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.

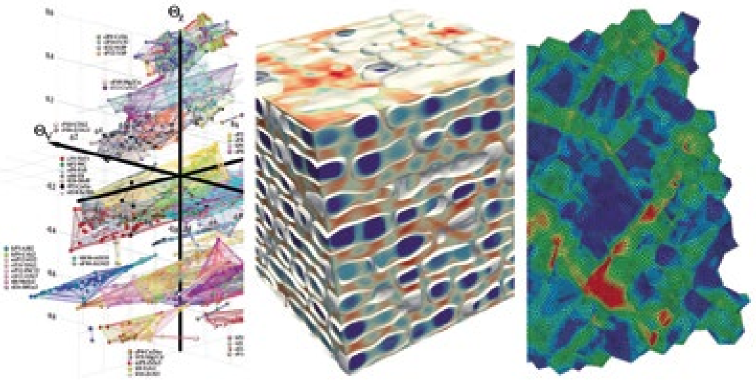

Fig. 1*: The three departments of ICAMS combine simulations at different scales, resulting into (from left) atomic structure maps, phase field and finite element simulations as typical images.*

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).

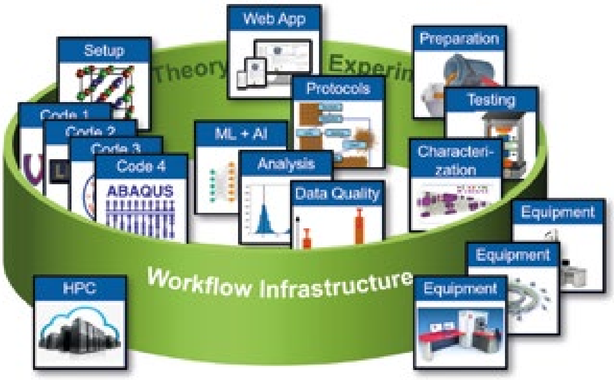

Fig. 2: *One of the main goals of PMD is to establish a workflow infrastructure that provides a user-friendly interface to a large variety of tools.*

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.

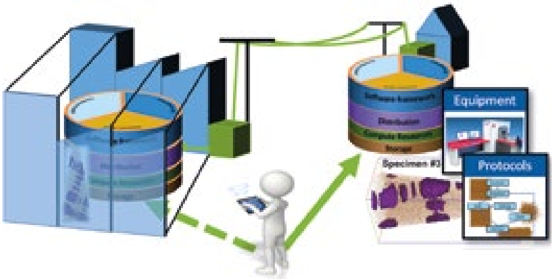

Fig. 1: *The decentralized data storage concept of the platform material digital, which supports the sharing of workflow solutions instead of huge amounts of raw data.*

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).

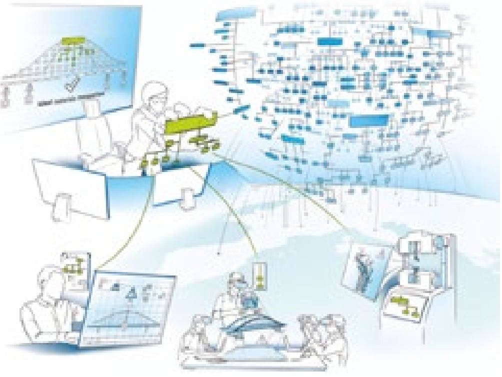

Fig. 1: *NFDI-MatWerk wants to make decentralized expertise integrable through a uniform data language and standardized interfaces.*

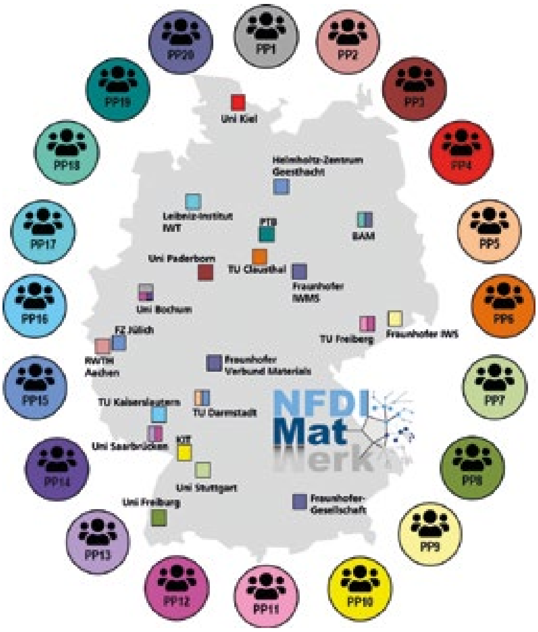

Fig. 2: *The geographic distribution of the participant projects (PP) of NFDI-MatWerk, which second the ten (co-) applicant institutions.*

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.

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.

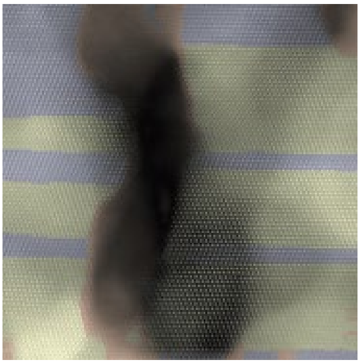

Fig. 1: *Automatically segmented HAADF-STEM image of Pd nanoparticles, highlighting twin domains (color-coded yellow, blue; red areas are featureless/amorphous).*

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.

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

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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.

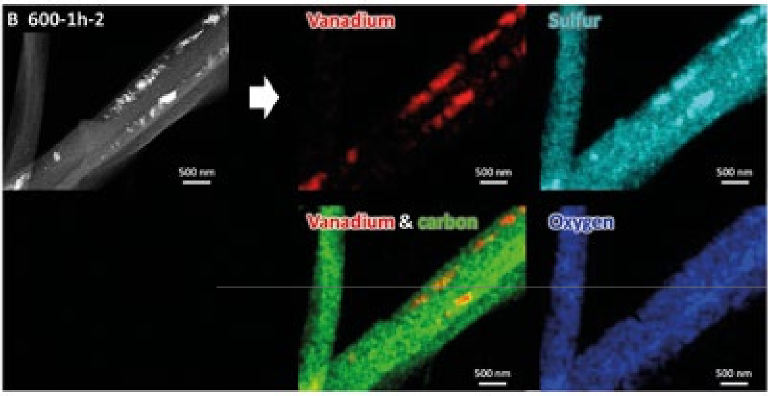

Fig. 1: *STEM image and corresponding elemental maps of vanadium, sulfur, carbon and oxygen showing the formation of vanadium sulfide particles embedded in carbon fibers. Images taken from [4].*

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].

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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).

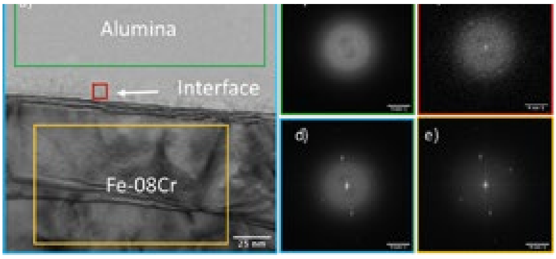

Fig. 1: *a) TEM micrograph of the film and substrate with and corresponding fast Fourier transformation data b) – e). The coating itself is amorphous while the interface shows mixed amorphous and crystalline parts.*

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.

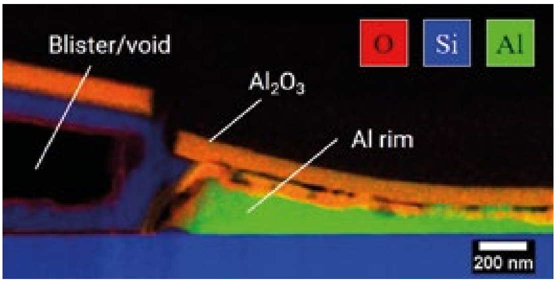

Fig. 2: *Cross-sectional STEM image overlaid with EDS elemental map reveals the microstructure change during annealing in high H_2 pressure. Blisters form with the initial amorphous Al_2O_3 layer remaining. A surrounding Al rim has built up due to dewetting. A part of the metallic film is transformed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

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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.

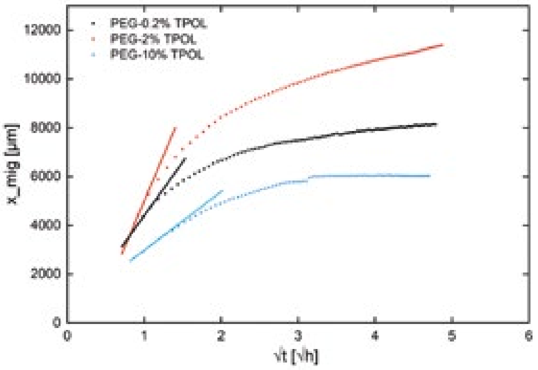

Fig. 1: *Progress of migration in humid nitrogen atmosphere for polyethylene glycol (PEG) coatings with different additions of TPOL: (2,4,6-(trimethylbenzoyl) diphenylphosphine oxide) initiator. Initially the migration rate is similar to the one of delamination (full lines).*

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.

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Cluster of Excellence “Ruhr Explores Solvation”

M. Rabe[1], M. Rohrwerder[1], M. Todorova[2], J. Neugebauer[2]

[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].

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).

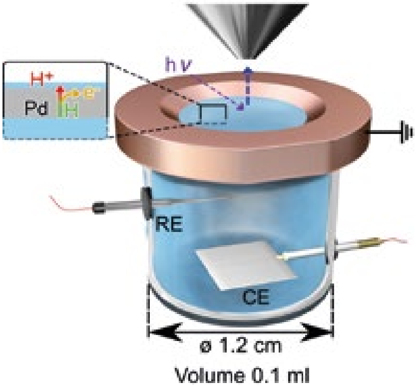

Fig. 1: *Experimental setup for an electrode in the dry for measurements with near ambient pressure x-ray photoelectron spectroscopy (XPS). The metal electrode is electrochemically charged with atomic hydrogen on the entry side. The hydrogen diffuses and sets a distinct potential at the dry, exit side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

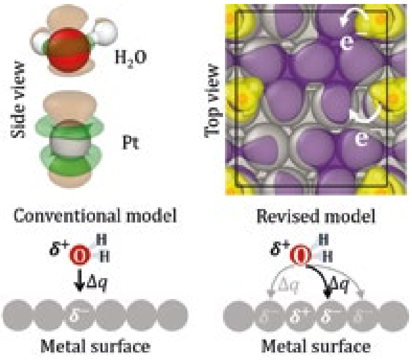

Fig. 2: *Our ab initio molecular dynamics calculations led to a revision of the well-established picture of water-metal bonding. The electron difference density (top) for the Pt(111)/water interface reveals the involvement of Pt atoms neighbouring the top water adsorption site (right). As consequence 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.

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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

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).

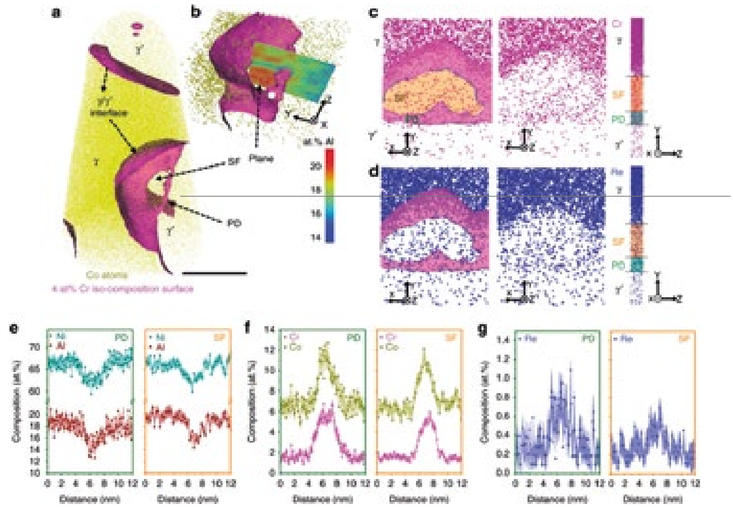

Fig. 1: *An APT reconstruction and analyses of the Re interactions with structural defects in a creep-deformed Ni-based alloy developed as part of the SFB TR103 (from [5], for details, see caption in the article).*

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.

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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.

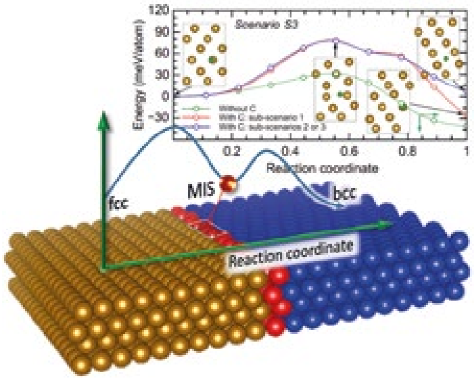

Fig. 1: *Ab initio determined minimum-energy path and impact of C on the structural evolution from a metastable intermediate structure (MIS) to the martensite. Figure adapted from [1].*

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).

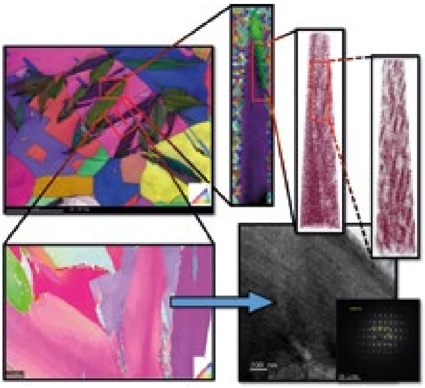

Fig. 2: *Correlative study TEM-APT analysis of a Fe-C microstructure containing austenite and martensite performed at RWTH Aachen University.*

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.

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Experiments for optimised CALPHAD modelling

B. Distl, Z. Kahrobaee, M. Palm, F. Stein

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].

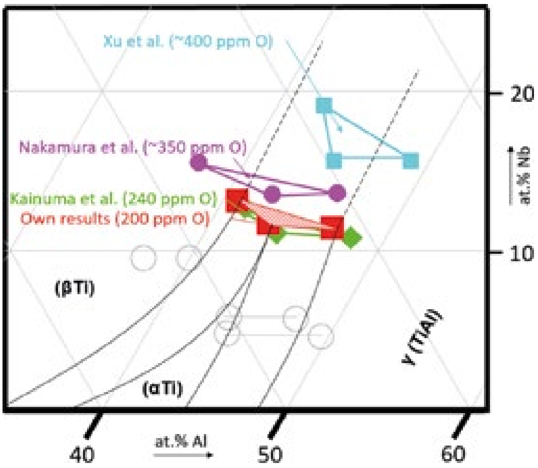

Fig. 1: *Partial isothermal section of the Ti–Al–Nb system at 1300 °C with experimental results from the literature and own results regarding the oxygen-related shift of the tie-triangle (αTi)+(βTi)+γ (TiAl) [2].*

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

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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].

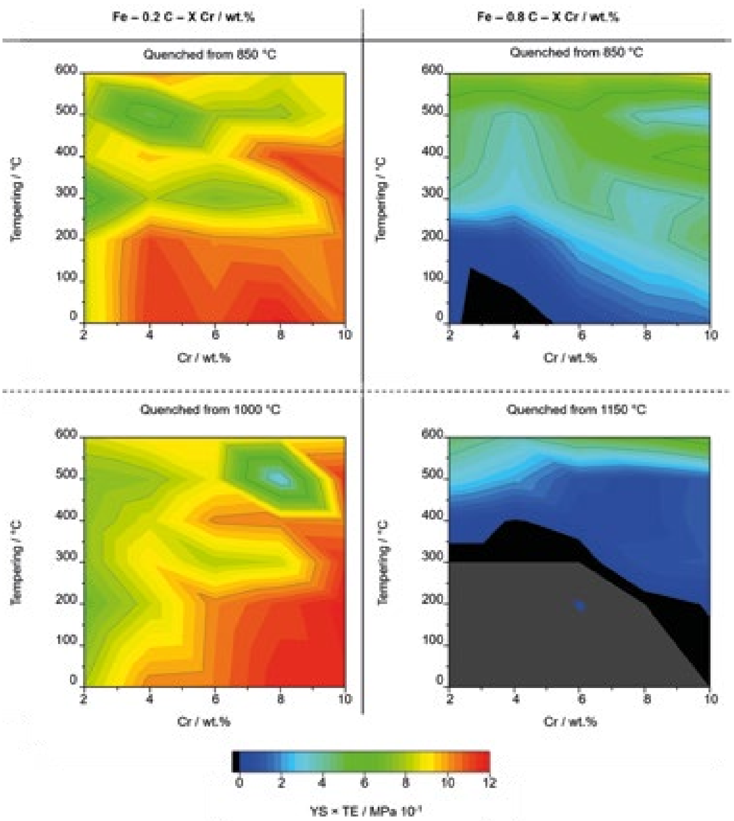

Fig. 1: *Heat maps displaying the property profiles of 100 states of Fe-Cr-C steels investigated with the combinatorial RAP methodology [4].*

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).

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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.

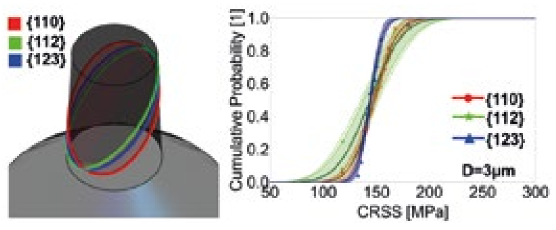

Fig. 1: *A Mathematica drawn ferrite pillar according to the electron backscatter diffraction (EBSD) orientation showing different slip traces distinguished by colour (left). Cumulative distribution of CRSS for slip plane families {110}, {112}, {123} (right) [1].*

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].

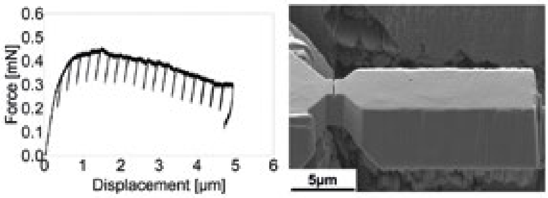

Fig. 2: *A representative force displacement curve for a beam showing crack growth during the cantilever bending test (left). Necked beam geometry contains martensite at the neck region (right). Martensite in this DP grade has a carbon content approximately 3 at.%. J-integral curve can be extracted [3].*

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.

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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.

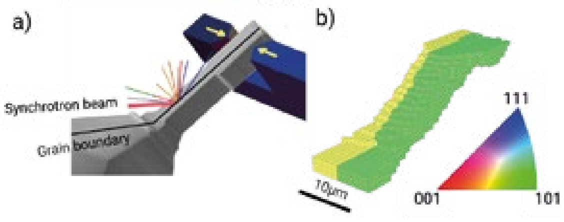

Fig. 1: *a) Schematic of the micro fatigue experiment on a single grain boundary. b) Reconstruction of the grain shape and grain orientation.*

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.

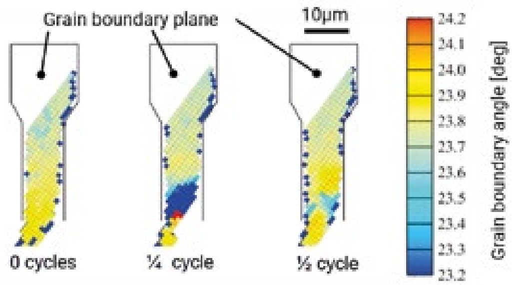

Fig. 2: *Grain boundary angle before loading, after ¼ and ½ cycles. Note the significant change in grain boundary angle after ¼ cycle, which is partly reduced during back-bending.*

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.

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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.

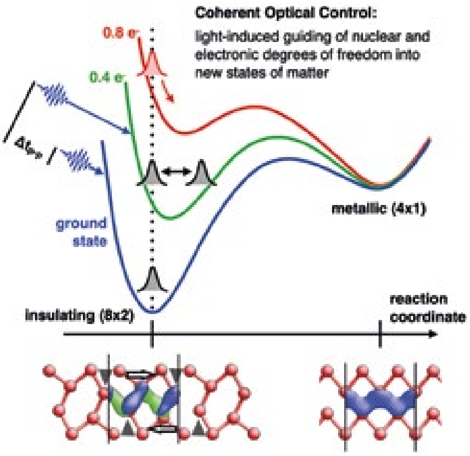

Fig. 1: *Ground and excited-state potential energy surfaces (PES) along a reaction coordinate, that transforms surface-supported indium atomic wires in an insulating CDW state into a supercooled metallic phase. Schematic structural models are shown below the PES, where red balls indicate indium atoms. 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.

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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.

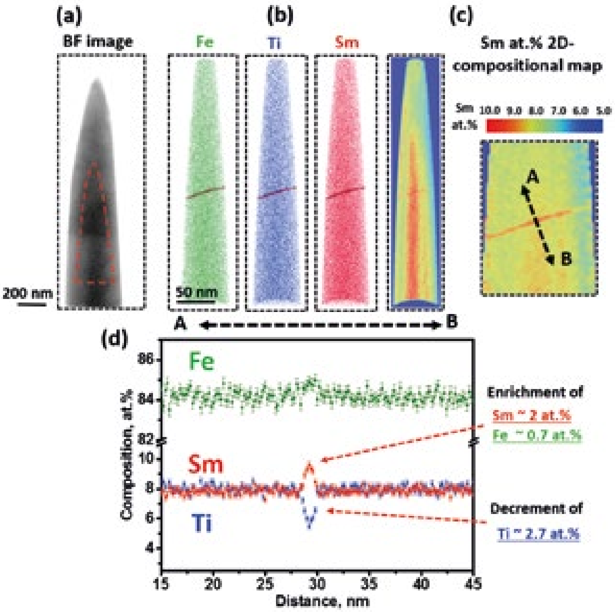

Fig. 1: *(a) Bright-field (BF) image of one of the investigated APT specimens. (b) Atom probe tomography reconstruction of the elemental distribution of Sm, Fe, and Ti. (c) 2D compositional map of Sm and (d) chemical composition distribution at the twin boundary region.*

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].

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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

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

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.

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.

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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.*

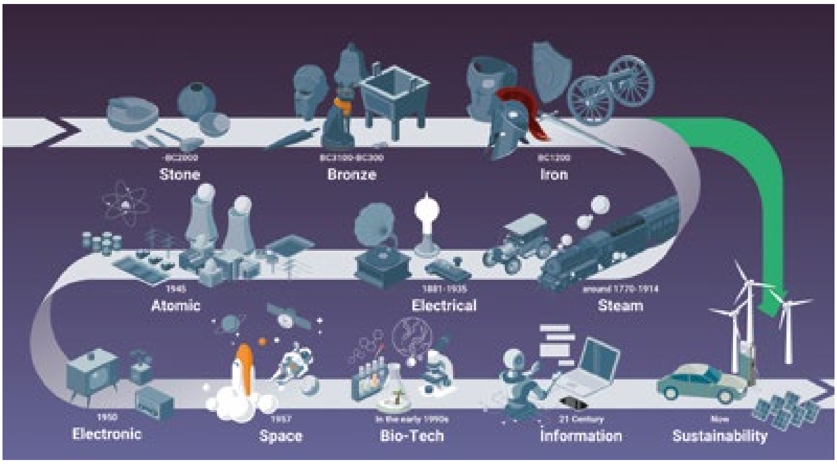

Fig. 4: *Developments in metallurgy are now more needed than ever. The picture is an example of the illustrations made by T. You, a graphic design student hosted by the PR.*

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 generally 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.

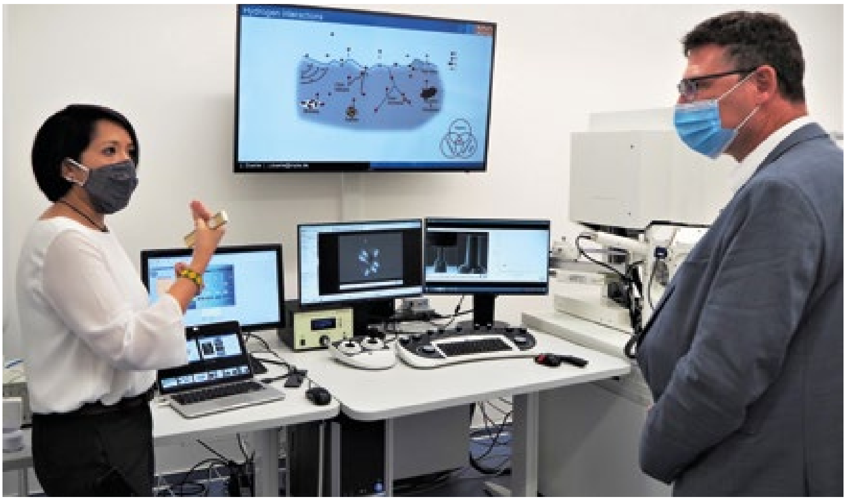
Fig. 5: *M.J. Duarte Correa is explaining her research on hydrogen resistant coatings to S. Kaufmann, delegate of the Federal Ministry for Education and Research in September 2020.*

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.