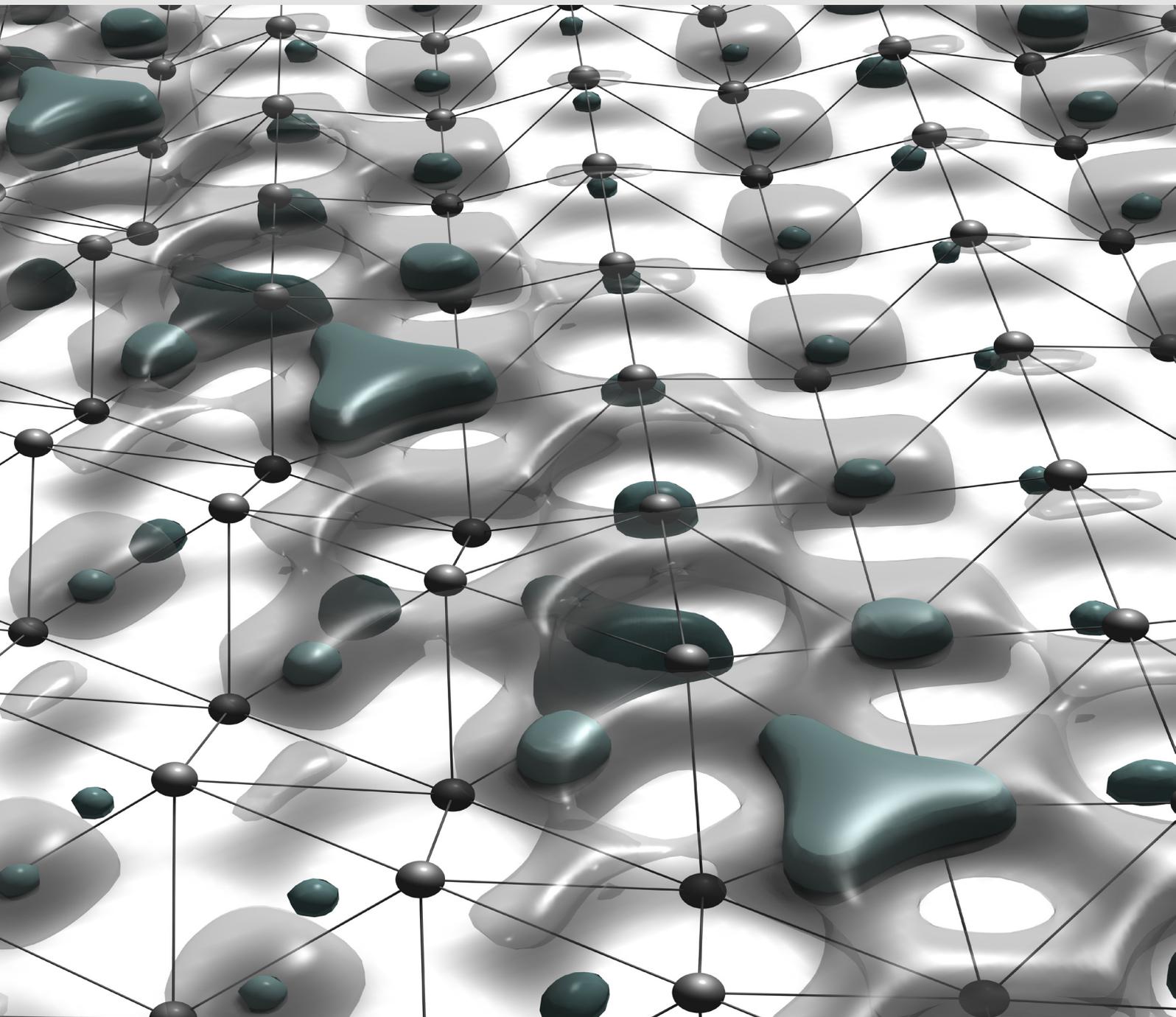




# Scientific Report 2007 / 2008

MAX-PLANCK-GESELLSCHAFT

Max-Planck-Institut  
für Eisenforschung GmbH



Max-Planck-Institut für Eisenforschung GmbH

# **Scientific Report 2007/2008**

December 2008

Max-Planck-Institut für Eisenforschung GmbH  
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Germany

### *Front cover*

Potential energy surface (PES) of an interstitial hydrogen atom in the vicinity of a symmetric tilt („twin“)  $\Sigma_{11}$  (110) grain boundary in nickel. The PES has been calculated in the CM department employing an analytical first-principles based potential. The PES provides direct insight into the adsorption energies and the kinetics of interstitial hydrogen: The green isoenergy surface marks regions of high binding energy, i.e., regions where hydrogen is trapped. Since these regions are significantly larger in the grain boundary than in the bulk region, the figure clearly demonstrates that the  $\Sigma_{11}$  (110) grain boundary acts as an efficient source of trapping hydrogen. This process may be regarded as an initial step for hydrogen-enhanced crack propagation, i.e., one of the main mechanisms causing hydrogen embrittlement of metals. The PES also provides information about hydrogen diffusion paths and barriers: possible low-energy diffusion paths are marked by the grey semi-transparent isoenergy surface.

For the visualization of the data the PHInaX program, developed in the CM department, has been used.

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# PREFACE

This report is part of a series summarising the scientific activities and performance of the Max-Planck-Institut für Eisenforschung. In particular, this volume covers the years 2007 and 2008.

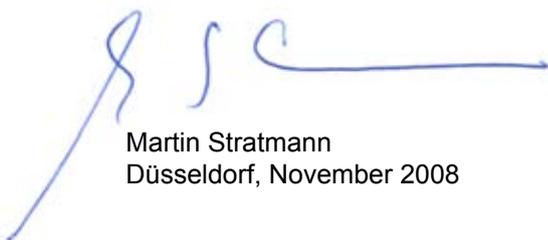
For the last two years all departments of the institute had been assigned to directors. This has not been the case for many years before as the institute has undergone a complete scientific reorientation. As a result the international visibility of the MPI für Eisenforschung has grown considerably. The institute now hosts 270 people, the majority of them being scientists. As only 120 employees are being paid by the shareholders of the institute, nearly 150 additional scientists could be attracted to work at the institute. As a result the MPI für Eisenforschung is one of the largest institutes devoted to fundamental research in steels and related materials.

Unfortunately Prof. Pyzalla recently decided to accept an offer as scientific general manager of the Helmholtz Centre Berlin for Materials and Energy. Furthermore Prof. Frommeyer will retire at the end of 2008 and therefore the institute again will have to reconsider its scientific focus and will have to attract internationally leading scientists at the directors' level within the near future.

The last years have seen new scientific groups and topics coming up and growing within the institute. The institute has further improved its network-like structure and established strong horizontal topics which also form its international reputation. Among those areas, scale bridging modelling of materials is probably most important and visible. All departments take part in this initiative and the institute has initiated scientific initiatives as well within the Max Planck Society as with the neighbour universities RWTH Aachen and Ruhr University Bochum. Within the institute, scale bridging modelling is strongly coupled to experiments, steel diagnostics and combinatorial materials research. Also the institute's industrial partners in particular within the steel industry are involved in these topics. Future developments will be aimed to strengthen this field further.

This report consists of the following parts:

- *Part I* deals with the organization of the institute including a short section on the recent developments as well as brief descriptions of the large-scaled and networking projects and some information on new scientific laboratories and facilities of the institute.
- *Parts II and III* cover the research activities of the institute. Part II provides a description of the departmental work and Part III contains selected papers which summarise major recent scientific achievements in the four areas of common interest of the institute *New Structural Materials*, *Microstructure-Related Materials Properties*, *Stability of Surfaces and Interfaces*, and *Scale-Bridging Simulation of Materials*.
- *Part IV* summarises the statistically relevant information of the institute.



Martin Stratmann  
Düsseldorf, November 2008



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# ***IN COMMEMORATION OF Prof. Dr. Dr.-Ing. E.h. mult. Hans-Jürgen Engell***



Hans-Jürgen Engell, emeritus scientific member of the Max-Planck-Institut für Eisenforschung, former director and general manager of the institute, passed away on September 20, 2007 at the age of 82. Hans-Jürgen Engell was born on 15th of October 1925 in Bad Reinerz, now part of Poland. He studied chemistry at the universities of Rostock and Greifswald where he did his doctorate on the chemisorption of gases on semi-conductors. His interpretation of chemisorption based on the Schottky defect model is still one of his most frequently quoted publications.

After his doctorate, he joined the Max-Planck-Institut für Eisenforschung in Düsseldorf and established one of the most outstanding international scientific groups in the field of corrosion research. As an excellent physical chemist, he published pioneering work in the fields of electrochemically controlled oxide dissolution, passivity, stress corrosion cracking and pitting corrosion. In these studies, he managed in a unique way to bridge the gap between the engineering science aspects of materials science and the underlying physical-chemical aspects of interface chemistry. Just one example: he was one of the first to apply impedance-spectroscopy as an electrochemical tool for the analysis of the properties of semi-conductors in aqueous electrolytes. This work, which was published in the journal *Zeitschrift für Elektrochemie* in 1957, had a strong influence on the application of this technique in corrosion research.

As the director of our institute since 1971, he pushed ahead the metallurgy of extremely low carbon, precipitation-hardened steels and initiated research projects for the quantitative calculation of phase transformations in steels. Under his direction the institute reached an internationally out-standing reputation, while he attended to the questions of corrosion research and metallurgy.

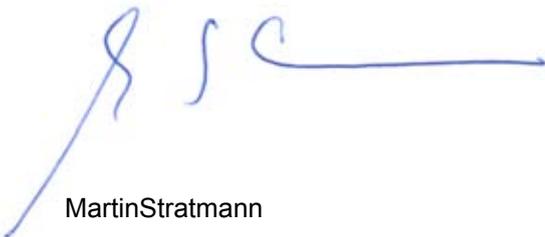
Hans-Jürgen Engell shaped and steered the corrosion research both in Germany and internationally; he was the initiator of a BMBF program on corrosion and corrosion protection, he was speaker of the DFG priority program in the field of corrosion research, and he was the chief editor of the journal "Werkstoffe und Korrosion" between 1968 and 1994.

As the support of young scientists was something he had especially at heart, he founded the Engell Award for talented young scientists which is awarded annually by the International Society for Electrochemistry and is now a highly reputable prize of this organization. His outstanding scientific work was confirmed by numerous awards. The German Society for Metallurgy awarded him with the Masing-Gedächtnispreis and subsequently with the Heyn-Denkmünze. The European Federation for Corrosion conferred him with the Cavallaro medal and the Institution of Corrosion Science and Technology of Japan made him an honorary member. He was then appointed a "Fellow of the American Society for Materials International" and in 1989 he became a foreign member of the Bulgarian Academy of Sciences. Further honorary memberships and honours were conferred in the year 1991 with the Dechema medal and the honorary membership to the "Société Française de Métallurgie et de Matériaux", Paris, while the Technical University in Berlin and the technical faculty at the University of Erlangen-Nürnberg awarded him with honorary doctorates in engineering.

Due to his teaching activities at universities in Clausthal, Stuttgart, and since 1977 in Düsseldorf, Hans-Jürgen Engell made a name for himself as a distinguished lecturer. Hans-Jürgen Engell also became strongly involved in science politics: he was the chairman of the University Structural Commission for the state of Lower Saxony and was a member of the Science Council for the Federal Republic of Germany between 1979 and 1985 becoming ultimately its chairman. His involvement in science policy was acknowledged in 1982 when he was awarded with a First Class Order of Merit of the Federal Republic of Germany.

His wide ranging knowledge and the liveliness with which he seized unconventional research subjects were exceedingly impressive. It is thus, that we express a grateful farewell to an important scientist and a warm-hearted, obliging and modest person. We will always preserve his honourable memory.

On behalf of the management board and all employees at the Max-Planck-Institut für Eisenforschung



Martin Stratmann

Chairman of the Management Board



## PART I.

# THE INSTITUTE

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# Management of the Institute

The Max-Planck-Institut für Eisenforschung (MPIE) is a joint venture between the Max Planck Society and the Steel Institute VDEh. Since half of the institute's budget is supplied indirectly through industry, this institute is unique within the Max Planck Society.

The institute was founded in 1917 by the Verein Deutscher Eisenhüttenleute (VDEh) and incorporated into the Kaiser Wilhelm Gesellschaft, the predecessor of the Max Planck Society. The institute 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 institute was integrated into the newly formed Max Planck Society in 1948. The institute 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. Until 2002, the institute was headed by

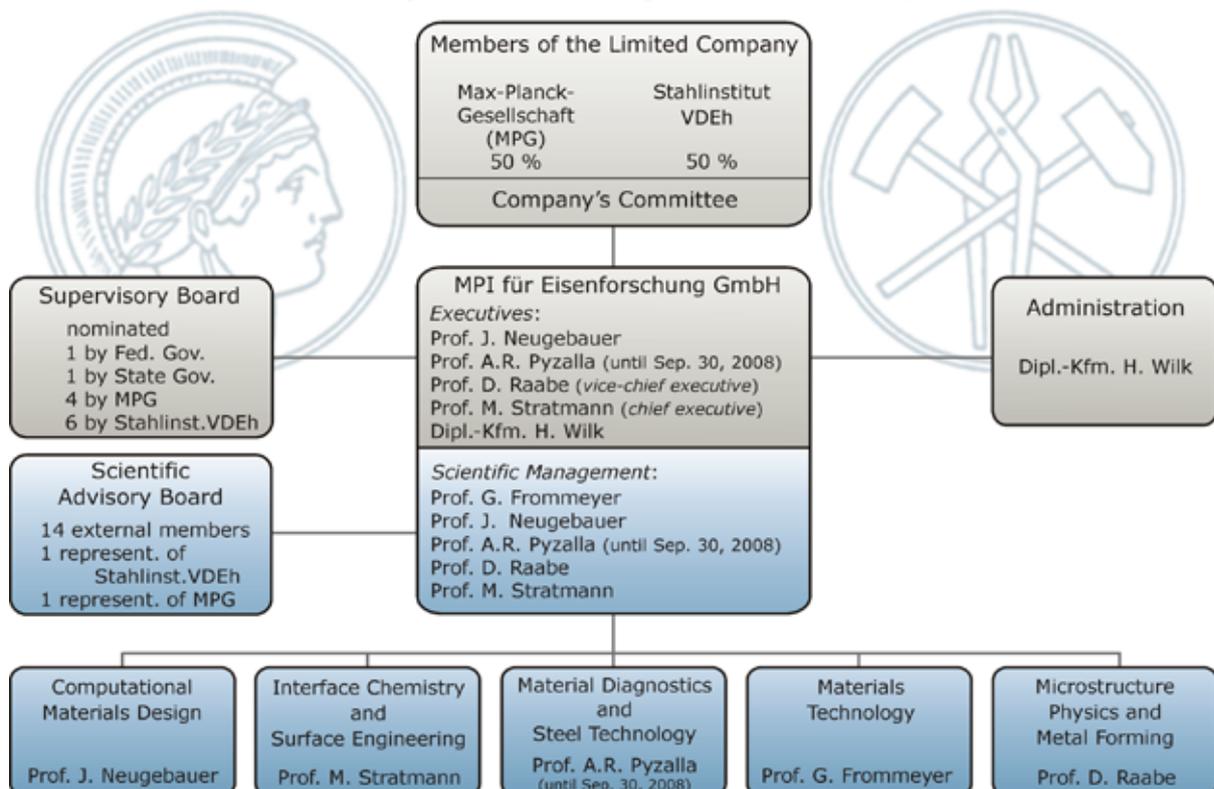
a chief executive director (1971-1990: Prof. Engell, 1990-2002: Prof. Neumann) and an associated administrative director.

Since June 2002, all scientific members of the institute form an executive board of directors. The position of a managing director will be filled, in rotation, by one of the board members. A board, which supervises the institute's activities, consists of representatives from the federal government, the state of Northrhine Westfalia, the Max Planck Society and the Steel Institute VDEh. A Scientific Advisory Board comprised of prominent scientists assists the institute in balancing fundamental research and technological relevance.

From 1971 until the present, the institute has operated on the legal basis of a limited liability company (GmbH) and its budget is equally covered by the Steel Institute VDEh and the Max Planck Society.

Since 2000, the institute again underwent refurbishment, which was completed in May 2008 (see also 'Recent Developments', p. 14).

## Max-Planck-Institut für Eisenforschung GmbH Management and Organization (2008)





# Scientific Organization

The institute devotes its research to iron, steel and related materials. In addition to the development of new materials, the institute focuses on the physical and chemical processes and reactions which are of importance for material production, processing, materials characterization and properties.

The institute is divided into the five following departments:

- *Computational Materials Design* (Prof. J. Neugebauer): description of materials properties and processing based on ab initio (parameter free) multiscale simulation techniques
- *Interface Chemistry and Surface Engineering* (Prof. M. Stratmann): aspects of environmentally accelerated degradation of surfaces and interfaces like corrosion and deadhesion and the engineering of new and stable surfaces and interfaces
- *Material Diagnostics and Steel Technology* (Prof. A.R. Pyzalla, until September 2008): microstructure - residual stress - property relations using e.g. electron microscopy,

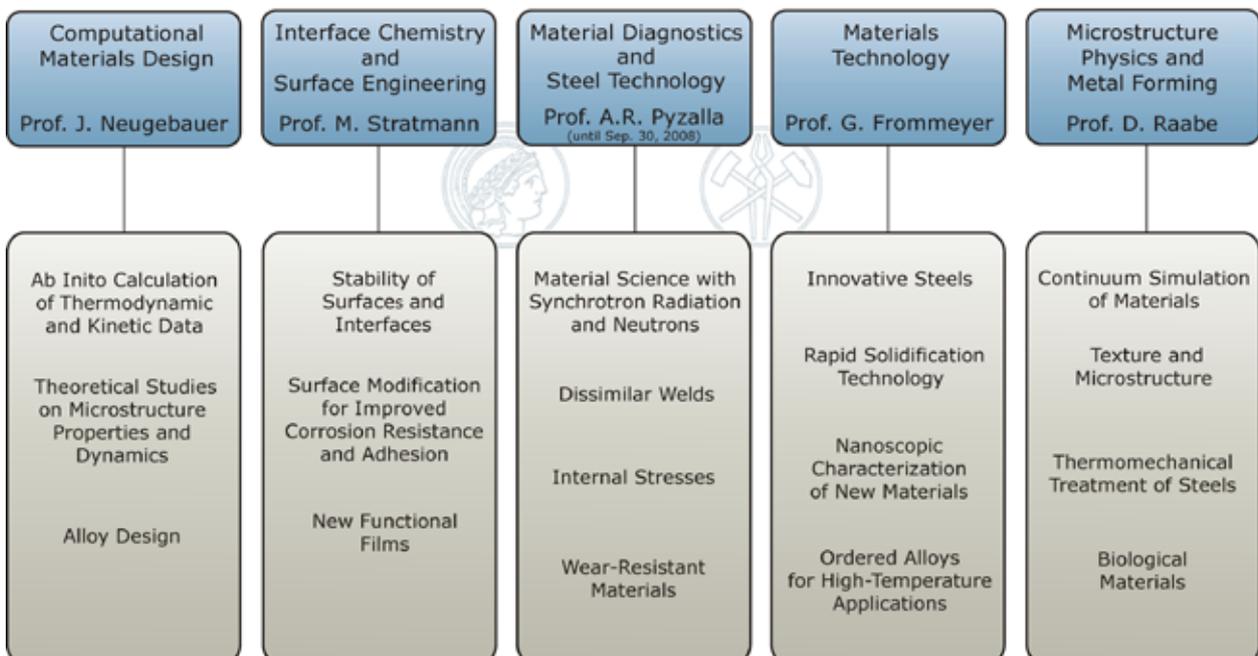
synchrotron and neutron diffraction, tomography and scattering.

- *Materials Technology* (Prof. G. Frommeyer): novel lightweight steels, new intermetallic materials for high-temperature applications and rapid solidification processes
- *Microstructure Physics and Metal Forming* (Prof. D. Raabe): mathematical modelling of microstructures and properties during processing and their experimental investigation using microscopy and diffraction methods

The main scopes of the departments are summarized in the figure below.

Each department is subdivided into research groups which are typically managed by group heads. The figure on the right side shows the organization of the groups within the departments. Each research group has its own specific focus and research activities. Part II of this report contains the summaries of the scientific concepts of the departments and brief descriptions of the research done in the different groups.

Scientific Scopes of the Departments





In addition to departmental research, certain research activities are of common interest within the institute. These central research areas are highly interdisciplinary and combine the experimental and theoretical expertise available in different departments. In concerted activities, scientific and technological breakthroughs in highly competitive research areas are achieved. Such inter-departmental research activities are described in Part III which is divided into the four topics

- New Structural Materials
- Microstructure-Related Materials Properties
- Stability of Surfaces and Interfaces
- Scale-Bridging Simulation of Materials

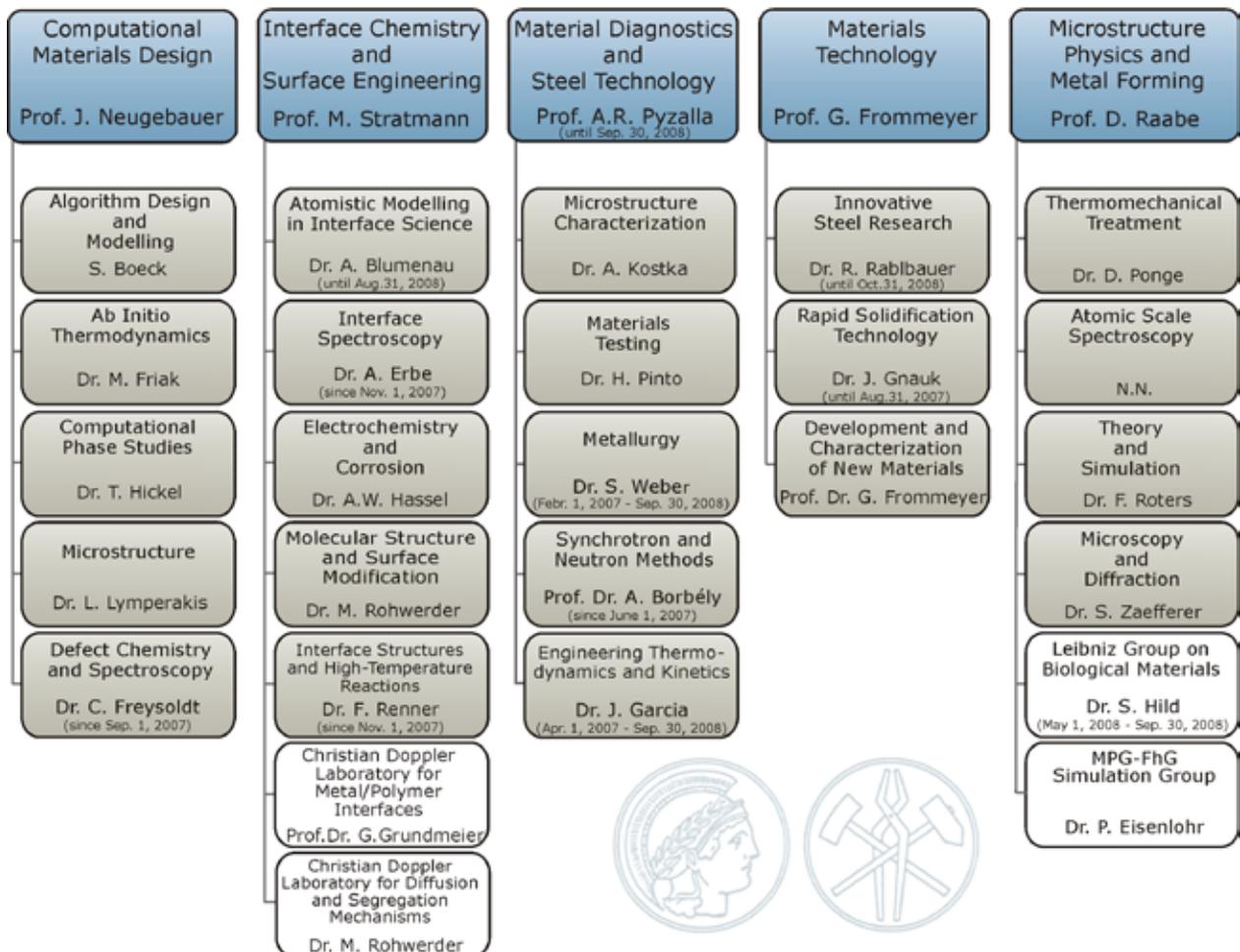
For each of these four central research areas, several short papers on selected scientific topics are provided in Part III giving an overview of the results obtained during the last two years.

In summary, the research within the institute is organized vertically in highly specialized departments

and research groups and horizontally in inter-departmental research activities. We believe that this form of organization encourages a high level of individual scientific work within the departmental framework of research groups as well as the development of new materials with complex properties combining e.g. high mechanical strength with high surface functionality. In a typical university setting, research activities such as metallurgy or surface science are carried out in different university departments. In contrast, these research activities are linked through the institute's research structure leading to a more efficient use of the scientific equipment and a homogeneous research profile.

Service groups provide the scientific departments with valuable experimental expertise. These services include the production of materials, chemical analysis of metallic substrates, metallography, a mechanical workshop equipped for the handling of unusually hard and brittle materials, facilities to build scientific equipment, an electronic workshop, a library and a computer network centre.

Scientific Groups within the Departments (2008)





# Recent Developments

In the reporting period 2007-2008 the institute has been working with a complete board of directors and an almost complete team of scientific group leaders for the first time since more than a decade. This together with an efficient and strategic use of the institute's resources resulted in an impressive amount of third-party funding and allows financing ~160 scientists including Ph.D. students and postdocs. The total number of employees at the Max-Planck-Institut für Eisenforschung GmbH currently stands at 270 making the institute one of the largest basic-science oriented steel research centers world wide.

In summer 2008, Prof. Anke Pyzalla received a highly prestigious offer to become a scientific executive of the new Helmholtz Centre for Materials and Energy in Berlin, a consortium of the former Hahn-Meitner-Institut and the synchrotron facility BESSY, which she accepted and therefore left the MPIE September 2008. The department "Materials Diagnostic and Steel Technology", which had been built up by Prof. Pyzalla starting in Nov. 2005 is therefore vacant again. Since also Prof. Frommeyer, heading the department "Materials Technology" retires by the end of 2008, the institute has started an intensive candidate search to close the gaps left by the closing of the two departments. In order to fill these gaps and at the same time match the future requirements regarding advanced diagnostics on the one hand and material synthesis on the other hand the institute's search extends particularly in these two fields.

After almost a decade of ongoing construction on essentially all buildings of the institute, the reconstruction and completion of the CM-building (the former "Blauer Bau") was a major milestone for the institute. It marks the completion of the extensive reconstruction which started in 2000 and which provides the institute now with first class infrastructure and scientific labs. The CM-building, which was finished in May 2008, is the new home of the department "Computational Materials Design" headed by Prof. Neugebauer and provides space for offices, the institute library, the network center and well equipped rooms for the next generation of the institute's high-performance computer cluster.

In the past two years a number of new scientific groups have been implemented: A second Christian

Doppler Laboratory within the department "Interface Chemistry and Surface Engineering" which covers a broad range of topics on diffusion and high temperature reactions under realistic production conditions for high strength steel strips (Dr. M. Rohwerder). Dr. Renner built up a group "Interface Structures and High-Temperature Reactions" which employs synchrotron radiation to investigate surface-gas and surface-liquid reactions on the atomic scale. Dr. Erbe has been appointed as head of the group "Interface Spectroscopy" which combines optical spectroscopy with advanced simulation techniques to study the structure of adsorbates and coatings in direct contact with a metal surface. In the department "Computational Materials Design" Dr. Freysoldt launched the group "Defect Chemistry and Spectroscopy" which extends and improves theoretical *ab initio* methods to follow the rapid progress in experimental spectroscopic techniques and to study defects of nonmetallic solids such as oxides or semiconductors. In the department "Microstructure Physics and Metal Forming" Prof. Hild opened a Leibniz group on biomimetic/bioinspired materials which studies e.g. titanium based medical implants or biological materials such as mineralized chitin. In the same department the formation of a new group and laboratory for 3D atom probe spectroscopy is currently in preparation. This technique will provide unique insights into the atomic structure and chemical composition of realistic alloys. It will be operated in close cooperation with the atomic scale simulation expertise in the CM department.

The combination of these newly established scientific activities with the already existing expertises significantly expands the profile of the institute and boosts further the in-house collaboration between the various departments and scientific groups. It also allowed building up new links and extending existing projects with various academic and industrial partners. Prominent examples which will be highlighted in the following are the DFG Collaborative Research Center (Sonderforschungsbereich) "Steel – *ab initio*", a joint research activity together with the RWTH Aachen (see p. 19), the foundation of the Interdisciplinary Center of Advanced Materials Simulation (ICAMS) at the Ruhr University Bochum (p. 17), or the Max-Planck Research School "SURMAT" (next page).



# Large-Scaled and Networking Projects

## International Max Planck Research School for Surface and Interface Engineering in Advanced Materials (IMPRS-SurMat)



R. Loschen, A.W. Hassel

**Introduction.** In 1999 the Max Planck Society together with the German Rectors' Conference (Hochschulrektorenkonferenz) and other Education Institutions in Germany launched an initiative to promote junior scientists called the International Max Planck Research Schools. In the winter semester of 2000/2001, the first Research Schools were started as cooperative efforts involving Max Planck Institutes together with German and foreign universities and research facilities. These schools offer outstanding students from Germany and abroad the possibility to prepare for their Ph.D. exam in a structured programme providing excellent research conditions.

**IMPRS-SurMat.** The concept of the International Max Planck Research School for Surface and Interface Engineering in Advanced Materials (IMPRS-SurMat) was approved by the Max Planck Society in 2003 and the first students took up their work in 2004. IMPRS-SurMat is a collaborative project involving the Max-Planck-Institut für Eisenforschung in Düsseldorf, the Max-Planck-Institut für Kohlenforschung in Mülheim/Ruhr, and four engineering and science departments of the Ruhr-Universität Bochum. The spokespersons of SurMat are Prof. Martin Stratmann and Prof. Gunther Eggeler. Members of the SurMat panel are the spokespersons and Prof. Martin Muhler (Ruhr-Universität Bochum) and Prof. Ferdi Schüth

(MPI für Kohlenforschung). The coordination office is placed in Düsseldorf. The administrative director (Dr. Rebekka Loschen) is the person responsible for all organisational issues such as application procedure, recruitment, public relations, troubleshooting, organisation of workshops and seminars etc. In this work she is assisted by Vanja Wüster. The scientific director, Dr. Achim Walter Hassel, takes care of all scientific issues linked to SurMat, as evaluation of research proposals or negotiating projects with industrial partners.

In addition to the aforementioned German partners Prof. Tian Zhongqun and Prof. Lin Changjian from the State Key Laboratory for Physical Chemistry of Solid Surfaces (PCOSS), Xiamen University, Prof. Mao Weimin from the Department of Materials, University of Science and Technology, Beijing, and Prof. Zhao Dongyuan from the Department of Chemistry, Fudan University are involved in this Research School. SurMat students working in cooperative projects with Chinese partners usually go to the corresponding Chinese institute for several weeks to perform experiments there. In the past the two students Juan Zuo (Grundmeier/Tian) and Ying Chen (Hassel/Tian) went to Xiamen for a research stay. Furthermore there is an active exchange of samples and measurements which results in joint publications.

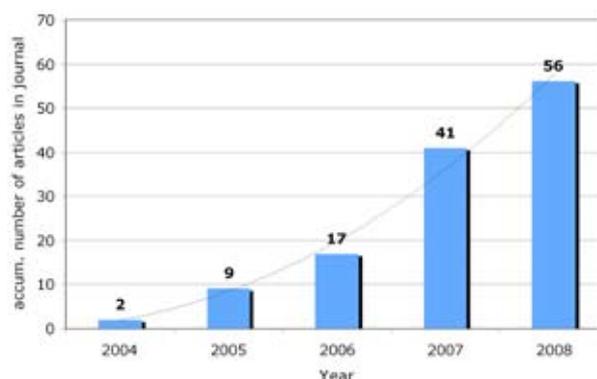
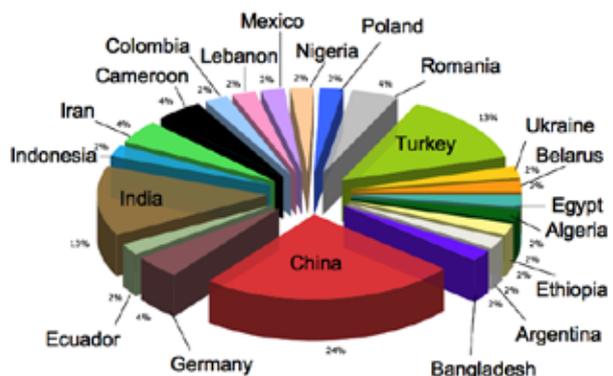


Fig. 1: Admission statistics by countries (left) and accumulated SurMat articles in scientific journals since 2004 (right).



Fig. 2: SurMat students during the workshop in Bochum, July 2008.

**Financing.** The major part of the SurMat funding is contributed by the Max Planck Society. Roughly 200.000 € are paid yearly to the school. As a start-up finance the government of North-Rhine-Westphalia supported SurMat with additional 200.000 €. Furthermore there are several industrial partners who sponsor special projects within SurMat. Companies such as ThyssenKrupp Steel AG, Chemetall GmbH and Salzgitter Mannesmann Forschung GmbH are valuable associates for SurMat. ArcelorMittal Eisenhüttenstadt GmbH and voestalpine Stahl GmbH have already agreed upon future cooperations and suitable candidates are presently being evaluated. The Ruhr-Universität Bochum contributes 60.000 € per year, of which 5 students can be financed.

**Status quo.** Since the start of IMPRS-SurMat in 2004, 44 students have been admitted to the programme. Eleven of them already have successfully finished their thesis, two even with distinction. Dr. Magdalene Walczak, now working at the DOC (Dortmunder OberflächenCentrum), has been awarded the Otto-Hahn-Medal, which is annually given to outstanding young scientists by the Max Planck Society. In the following few months approx. ten more students will be finishing their work. For this reason recruitment of new potential candidates is one of the major tasks of the coordination office. In the past few years more than three quarters of all applicants came from India, Iran or China. Despite this fact, the „nation's cake“ of SurMat is well mixed (Fig. 1).

For an even more international recruitment it is planned to cooperate with branch offices of the DAAD all over the world, especially in South-American and East-European countries, which only have been little attracted by SurMat up to now.

The productivity of the SurMat members can well be seen in Fig. 1. In the year 2007, 24 articles

in internationally renowned journals have been published, presumably the number is increasing for the year 2008.

**Latest events.** The years 2007/08 were quite decisive for SurMat as many important events took place. First of all, there is new staff at the coordination office with Rebekka Loschen as the administrative director and Achim Walter Hassel as the scientific director. The new team started off by organising two successful events. From 15-16 July 2008 the 2nd International SurMat workshop took place at the Ruhr-Universität Bochum. During these two days, SurMat students and members as well as industrial partners could follow a most interesting programme, held by guest speakers and SurMat students. Place and time were given for fruitful interdisciplinary scientific discussions.

The second event was the SurMat seminar in Kleve, from August 12-15, 2008. During these four days all SurMat students were encouraged to give presentations on their research, which subsequently were discussed lively by the other participants. The location, Schloss Gnadenenthal, is situated outside of Kleve, near to the home of Joseph Beuys, the famous artist. In this calm and picturesque surrounding the SurMat group could not only discuss scientific topics but also got to know each other better. An extensive discussion round between the coordinators, supervisors and students gave important impulses for SurMat concerning future amendments and targets.

**Outlook.** The first 6-year period of SurMat will be ending soon in 2009. The main target of the SurMat board is now to extend the programme for another 6 years. Therefore, SurMat will be evaluated in spring 2009 and preparations for this very important event are already being made.



## Interdisciplinary Centre for Advanced Materials Simulation – ICAMS

*J. Neugebauer, T. Hickel,  
A. T. Blumenau, M. Stratmann*

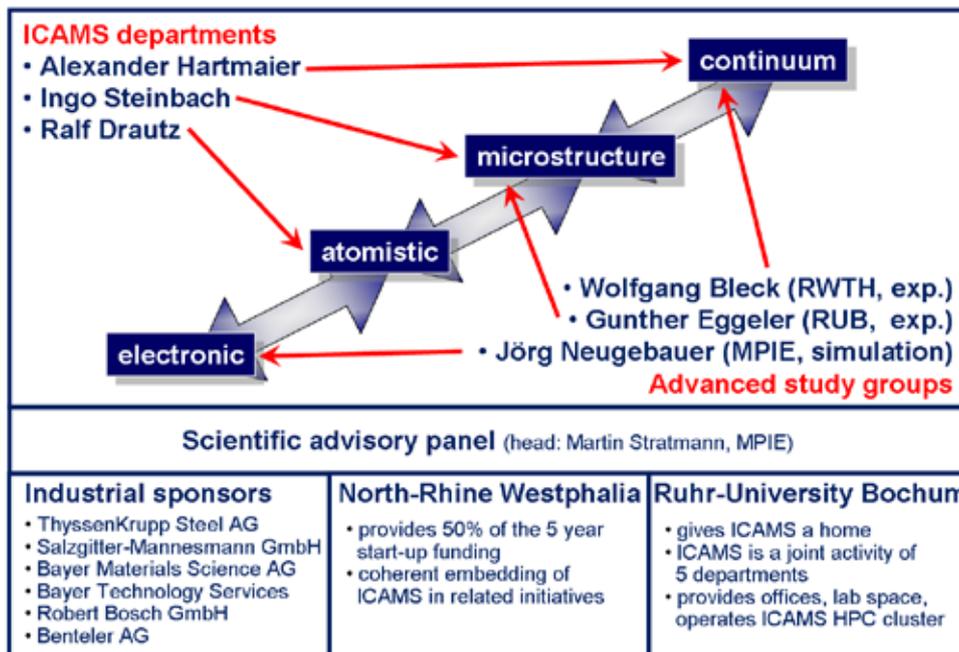


“Advanced Materials Simulation”, an interdisciplinary field of materials science and engineering, is increasingly gaining attention. This field has its roots in physics, chemistry, the materials sciences and other engineering branches and strong connections to mathematics and computer science. The availability of state-of-the-art computational simulation tools and of experts in this field will be crucial to meet the ever growing industrial demand of novel materials with tailored properties and efficient fabrication processes. To strengthen the long-standing expertise in advanced materials in the Rhine-Ruhr area and to boost the already existing expertise in materials simulation the Ruhr Area Initiative chaired at that time by the ThyssenKrupp Steel AG and the Max-Planck-Institut für Eisenforschung (MPIE) proposed and developed the concept of a Interdisciplinary Centre for Advanced Materials Simulations.

In March 2007 ICAMS was founded at the Ruhr-University Bochum (RUB). It is supported by a consortium led by ThyssenKrupp Steel AG, Salzgitter-Mannesmann Forschung GmbH, Robert Bosch GmbH, Bayer Materials Science AG and Bayer Technology Services GmbH, Benteler AG together with the MPIE, the RWTH Aachen University and the state of North Rhine Westphalia. The total financial support of 24.2 million Euros for the five years starting phase is provided by industry and the State of North Rhine Westphalia in equal shares.

The objective of ICAMS and its partners like the MPIE is to develop the next generation of simulation tools for materials modelling and to transfer these simulation tools into industrial research and product development. As materials with specific properties become increasingly important for the development of new products, the goal of ICAMS is to guide the design of novel materials using computer simulations. A field where ICAMS and its partners will become active is the development and understanding of high strength steels as required by the car industry for weight reduction and reduced fuel consumption while simultaneously increasing passenger safety. Another field is the development of simulation tools to design new and reliable high-temperature alloys for energy companies to increase thermal efficiency.

Since engineering materials have a complex and hierarchical microstructure extending over several orders both in length and time scale, a key objective of ICAMS is to develop and apply modern multiscale approaches which allow to bridge between scales. Based on these considerations ICAMS is organized in a matrix structure in which the scientific expertise of three departments and three advanced study groups will be combined under the direction of leaders in their fields while research projects will be conducted horizontally, across departments. The departments are directed in the five-year start up phase by endowed chairs which will later





be integrated into the Ruhr University. ICAMS succeeded in appointing internationally renowned experts in the fields of atomistic (Prof. R. Drautz, formerly Oxford), microstructure (Prof. I. Steinbach, formerly ACCESS Aachen) and continuum (Prof. A. Hartmeier, formerly University of Erlangen) simulation. These three central ICAMS departments are complemented by three external Advanced Study Groups, which link ICAMS to the fields of expertise within the MPIE, the Steel Department of the RWTH Aachen (*Institut für Eisenhüttenkunde*) and the experimental materials science groups of the RUB. The corresponding groups are headed by Prof. J. Neugebauer (simulation), Prof. G. Eggeler (Experimental Input and Validation) and Prof. W. Bleck (Manufacturing and Processing).

This combination of expertise at all scales in a single institution promises a hitherto not achievable integrated simulation of engineering materials and a predictive power which is also internationally unique. The resulting simulation tools will provide the benefit of predicting the behaviour of components, both during manufacture and when in operation, without the need for complicated experiments, from a comprehensive parameter-free description of materials on the atomic level (*ab initio*), through the mesoscopic or microstructural scale, to the macroscopic component behaviour. Using these methods, complex industrial problems can be solved

arising in the areas of development, manufacturing and the application of materials. The close proximity of ICAMS to major industrial research centres will enable a close connection and collaboration with industry and will further strengthen the high materials competence in the Rhein-Ruhr area. A key task of ICAMS is also to educate scientists in this new and emerging field. Therefore, innovative master and doctoral courses are presently built up and use the unique interdisciplinary ICAMS structure. These courses will not only embrace mechanical engineering with its materials science focus, but also include in particular the other engineering fields, as well as chemistry, physics, and mathematics. As a result a new generation of materials engineers will be qualified with broad interdisciplinary expertise, in particular in the field of materials simulation.

The MPIE is strongly involved and supports these activities. There has been an international conference on "Ab initio description of Iron and Steel (ADIS2008)" jointly organized by the MPIE-CM department and ICAMS at Schloss Ringberg. Further, in September 2008 a workshop on multiscale simulations has been organized by the MPIE-CM department in Ebernburg where also all ICAMS directors and many of the ICAMS scientists participated. These meetings together with smaller meetings between individual departments and groups initiated a number of joint ICAMS-MPIE projects.



## Steel – ab-initio: Quantum-Mechanics Guided Design of New Fe-based Materials

*D. Raabe, J. Neugebauer, F. Roters, S. Zaeferrer, M. Winning, T. Hickel, H. Pinto*



**RWTH**

In 2007 the Max-Planck-Institut für Eisenforschung in Düsseldorf (MPIE) and RWTH Aachen University jointly initiated a new collaborative research center (Sonderforschungsbereich, SFB 761) on the quantum mechanics guided design of new Fe-based materials. The initiative is funded by the Deutsche Forschungsgemeinschaft DFG (German Research Foundation, www.dfg.de). Speaker and chairman of the project is Prof. Wolfgang Bleck (Institute of Ferrous Metallurgy, RWTH Aachen University) and vice-chairman is Prof. Dierk Raabe (MPIE).

Scientists from three departments of the Max-Planck Institute are involved in 4 projects of the initiative which consists of altogether 16 projects organized in 3 research fields (Theory, Processing, Characterization), Figs. 1 and 2. The assignment is the development of new iron-based materials on the basis of ab-initio predictions in conjunction with advanced characterization and metallurgical alloy development tools. The aim is to establish a new ab-initio-based approach for the time- and cost-efficient development, understanding, and characterization of new engineering materials.

The first phase of the project concentrates on the ternary Fe-Mn-C system which forms the basis of high manganese steels. Such alloys are prominent examples of next generation high strength steels which exploit the TRIP and the TWIP effects for excellent forming and strain hardening characteristics (TRIP: transformation-induced plasticity; TWIP: twinning-induced plasticity), Fig. 3. Furthermore, the Fe-Mn-C system is well suited for this project since the thermodynamics and mechanical properties of this class of structural materials are influenced by the chemical composition in a sensitive manner. Already at an early stage of the project ab-initio calculations provide thermodynamic properties (such as the structure, stacking fault energies, elastic properties) that can be used in phenomenological models for optimized alloy design. Better understanding of the fundamentals of thermodynamic, kinetic, and mechanical mechanisms are crucial for exploiting the extraordinary potential in terms of strength and ductility of high manganese steels. Experimental validation and process technology development are carried out parallel to the theoretical parts of the project, Fig. 1.

A: Theory	B: Processing	C: Characterization
<b>A1</b> Dronskowski: ab-initio quantum chemistry	<b>B1</b> Senk: alloying, casting, solidification	<b>C1</b> Mayer: microstructure analytics
<b>A2*</b> Neugebauer/Hickel: ab-initio thermodynamics	<b>B2</b> Hirt: warm deformation processing	<b>C2</b> Bleck: deformation mechanisms
<b>A3</b> Schneider/Hallstedt: thermodynamics	<b>B4</b> Molodov/Gottstein: heat treatment	<b>C3</b> Schneider/Music: local mechanical properties
<b>A4</b> Emmerich: phase field theory		<b>C4*</b> Raabe/Zaeferrer: texture and anisotropy
<b>A5</b> Bleck: plasticity mechanism maps		<b>C5*</b> Pinto: defects and internal stresses
<b>A6</b> Gottstein/Mohles: interfaces		<b>C6</b> Prah: damage and defects
<b>A7*</b> Roters/Winning: micro-structure mechanics		

Fig. 1: Structure of SFB 761. A2, A7, C4, and C5 are MPIE projects.



Fig. 2: Scientists collaborating in SFB 761.

The long-term targets of SFB 761 are:

- Development of a new approach to material- and process-design based on ab-initio calculations.
- Rapid development and maturation of new engineering materials through efficient “scale-

hopping“ of theoretical results among relevant time and length scales.

- Material-Design of a new class of structural materials based on the Fe-Mn-C system.

The short term aims are:

- Manufacturing and characterization of Fe-Mn-C-steels of different compositions.
- Use of ab-initio methods for the prediction of phase transitions and mechanism changes (e.g. TWIP, TRIP, shear band formation).
- Quantification of the effect of chemical composition and temperature on the occurrence of different strengthening mechanisms.

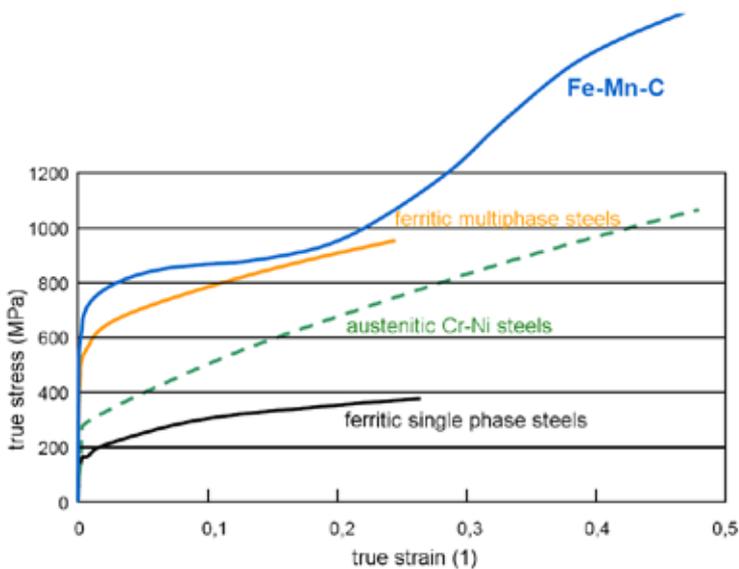


Fig. 3: Typical true stress – true strain flow curves of different classes of steels.

More details on SFB761 are on <http://stahl-abinitio.de>.



## Triple-M – Max Planck Initiative on Multiscale Materials Modelling of Condensed Matter

*D. Raabe, J. Neugebauer, O. Dmitrieva, M. Friák, M. Petrov,  
J. von Pezold, S. Nikolov, A. Counts, D. Ma*

In 2006 the Max Planck Society launched the “Max Planck Initiative on Multiscale Materials Modelling of Condensed Matter- Triple-M” as an interdisciplinary initiative among 6 Max-Planck institutes on the theory of solid matter.

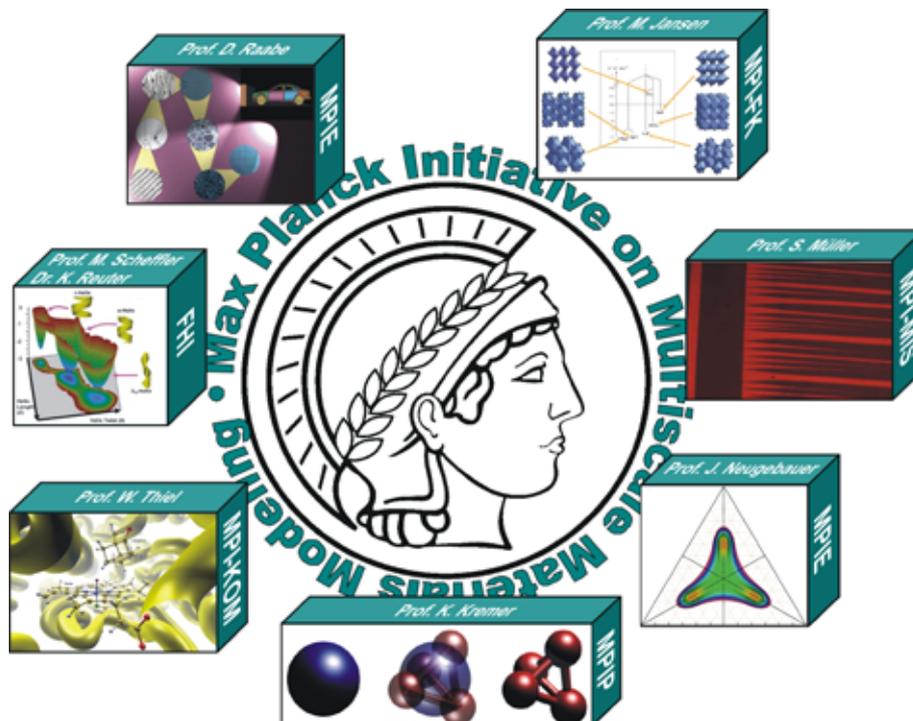
The initiative brings together different condensed state theory groups to develop and apply novel simulation tools for bridging time and space scales in materials science modelling. The methods employed range from quantum chemical to continuum modelling methods. Details on the structure and overall contents of the MMM initiative were outlined in the preceding scientific report.

Principal investigators are Prof. Martin Jansen (MPI for Solid State Research), Prof. Kurt Kremer (MPI for Polymer Research), Prof. Stefan Müller (MPI for Mathematics in the Sciences), Prof. Jörg Neugebauer (MPI für Eisenforschung, MPIE), Prof. Dierk Raabe who is the initiator and speaker of the

initiative (MPIE), Prof. Matthias Scheffler (Fritz-Haber Institute), Prof. Walter Thiel (MPI for Coal Research), and Dr. Karsten Reuter (Fritz-Haber Institute).

A first joint Max-Planck Multiscale Simulation Conference was organized by MPIE in St. Feliu de Guixols, Spain (28. Oct.-2. Nov. 2007) involving about 50 participants from all groups.

In this report we present three joint projects which are funded under the umbrella of the MMM initiative. The first one is on the use of ab initio simulations based on density functional theory for the design of body centered cubic magnesium-lithium for ultra-light weight engineering applications (Counts et al., p. 139). The second one is about the understanding of micro-band patterning phenomena in crystal plasticity (Dmitrieva et al., p. 183) and the third one deals with the scale-bridging simulation of chitin-based biological nano-composites (Nikolov et al., p. 169).





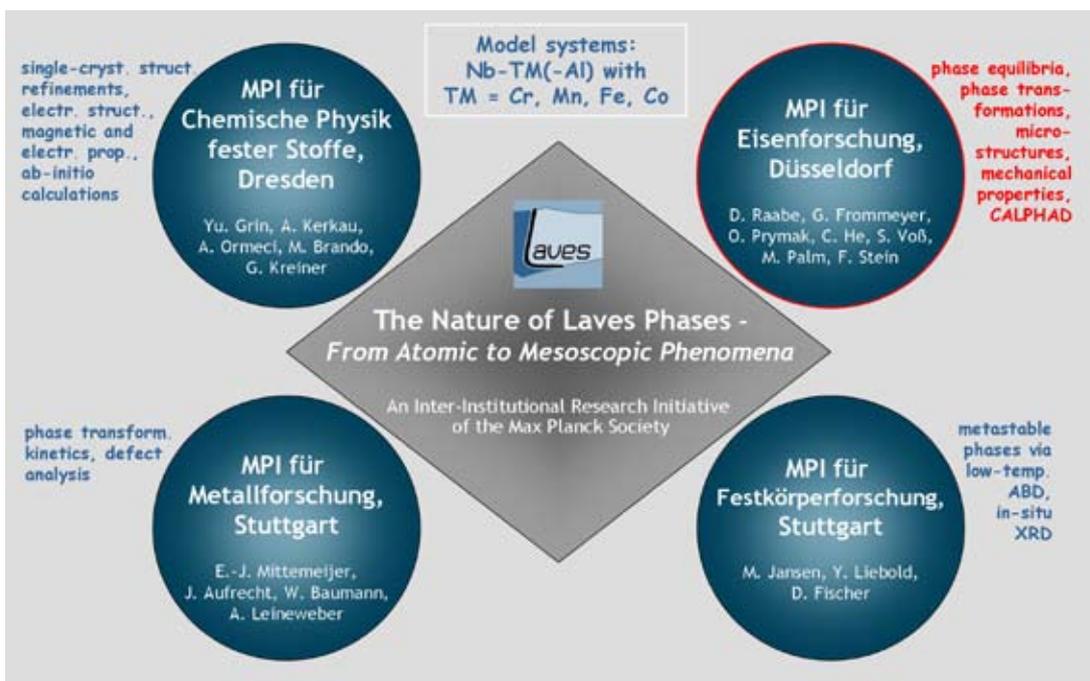
## The Nature of Laves Phases – An Inter-Institutional Research Initiative of the Max Planck Society

F. Stein, O. Prymak, S. Voß, C. He, M. Palm, G. Frommeyer, D. Raabe

The inter-institutional research initiative “The Nature of Laves Phases”, which is funded by the Max Planck Society since 2006, is a co-operation of four Max Planck institutes. The MPI for Chemical Physics of Solids in Dresden, MPI for Metal Research and MPI for Solid State Research, both in Stuttgart, and the MPI für Eisenforschung (MPIE) are closely collaborating within this research initiative in order to create a high-level forum with strong interdisciplinary character for the experimental and theoretical investigation of complex intermetallic phases. The joint scientific expertise and research equipment of the involved Max Planck research groups promise a breakthrough in the understanding of intermetallic phases ranging from atomic to mesoscopic phenomena. The scientific topic is comprehensive and includes synthesis of novel, high-melting phases, investigation of their thermodynamic stabilities and crystallographic structures, determination of physical properties, quantum mechanical and thermodynamical modelling of the phases as well as studies of corresponding kinetic phenomena. The Laves phases, which form the largest group of intermetallic phases, are chosen as model systems. The physical metallurgy of the intermetallic phases is much more complex than that of other established alloys, but has to be understood in greater detail as these phases are promising candidates for the design of new metallic materials

with the intermetallic phase as the matrix phase or as hardening precipitates, e.g. in high-performance steels.

Over the last two years the MPIE is working very actively within this project as is demonstrated not only by various presentations at international conferences [1-26] and journal publications [27-31] but also by the organization of international workshops on this subject [32,33] and several co-operations with international experts from the USA, Japan, China, and Europe. During August 2007 and June 2008, Prof. S. Kumar from Brown University (Providence, RI, USA) visited the MPIE for two guest stays. Within this ongoing collaboration, the microstructural evolution and phase transformations in Laves phase alloys are studied by in-situ transmission electron microscopy [11,26]. Other co-operations are with Prof. J.C. Schuster from the University of Vienna, Austria (experimental determination of phase diagrams and their thermodynamic description with the CALPHAD method; stay at the MPIE in October 2008) [34], Prof. M. Takeyama from the Tokyo Institute of Technology, Japan (phase relations of Laves phases in the Cr-Ti system; stay of his doctoral student S. Ishikawa for 6 months at the MPIE) [9,10,13,14], Prof. Yong Du from the Central South University in Changsha, China (CALPHAD modelling of the ternary Nb-TM-Al





phase diagrams with TM = Cr, Co, and Fe; stay of his postdoc Dr. Cuiyun He for one year at the MPIE) [21], and Dr. D. Daloz from the Ecole des Mines de Nancy, France (thermodynamic assessment of the Fe-Al-Zr system).

Currently, four different research projects on Laves phases are in progress at the MPIE which are briefly described on p. 101/102 in Part II of this report.

More information on the research initiative "The Nature of Laves Phases" can be found on the project website <http://laves.mpie.de>.

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## Aachen Institute for Advanced Study in Computational Engineering Science (AICES)

P. Eisenlohr, M. Friák, F. Varnik,  
D. Raabe, J. Neugebauer



**Introduction.** The Aachen Institute for Advanced Study in Computational Engineering Science (AICES) is a graduate school that has been established within the framework of the Excellence Initiative of the German federal and state governments in 2006. The Max-Planck-Institut für Eisenforschung GmbH is, together with the Research Centre Jülich, the leading academic partner of RWTH Aachen in this initiative (for details see [1]).

**Mission.** The AICES program is designed to provide a thorough training at the interface of classical engineering, materials science, applied mathematics, and computer science. The focal issues are interdisciplinary and method-oriented challenges within the simulation-based engineering science, as e.g. multiscale problems bridging the gap between the atomistic and macroscopic properties and phenomena. The AICES Graduate School complements and enhances the existing research activities at both RWTH Aachen and MPIE.

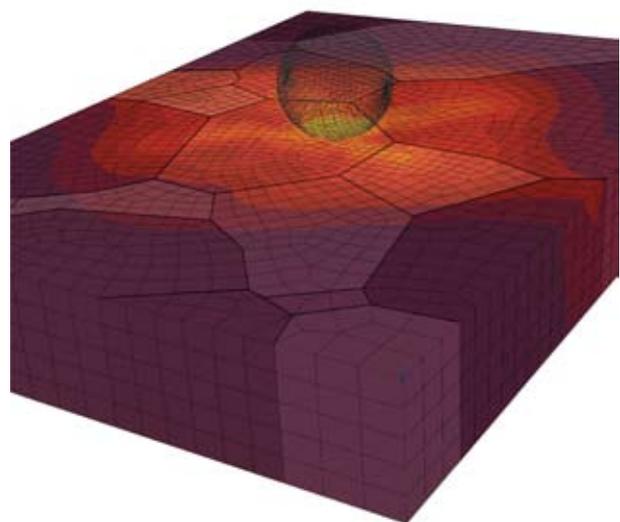
Dr. Philip Eisenlohr (MU department of Prof. D. Raabe) and Dr. Martin Friák (CM department of Prof. J. Neugebauer) became junior research group leaders associated with the AICES program during 2007, Dr. Fathollah Varnik (MU department) has become one of the AICES senior scientists.

**Lecturing.** In 2008 Dr. Eisenlohr held a series of lectures on “*Theory and Applications of a Computational Framework for Crystal Plasticity*” within the 3<sup>rd</sup> trimester of the European Union Regional School “*Materials Science*” that is open to graduate students from German, Belgian and Dutch Universities. One of the lectures, “*FEM: a basic overview of the method & outlook on applications*” was given by Dr. A.W. Counts (MU department), and another, “*Ab initio based approaches to the theory-guided materials design*”, by Dr. Friák (CM department). As a part of networking activity between the AICES and other institutes at the RWTH Aachen, Dr. Varnik gave in the years 2007 and 2008 two series of lectures at the Institut für Metallkunde und Metallphysik (IMM). The first series of lectures were on “*Lattice Boltzmann simulations of non-ideal fluids*” (winter semester 2007/08) and the second series on “*Computer-aided modelling of wetting phenomena: Theory and application*” (summer semester 2008). In winter semester 2007/2008, Dr. Varnik also held

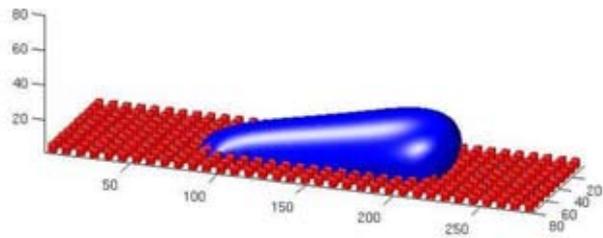
two lectures on the lattice Boltzmann method as part of the lecture series on “*Materials Modelling*” given by Prof. Heike Emmerich at the Institut für Gesteinshüttenkunde (GHI).

**Research topics.** Dr. Eisenlohr’s research activities in connection with AICES deal mostly with aspects of interfaces in crystal plasticity. An important question, which has to date not satisfactorily been answered, is the incorporation of slip resistance exerted by random high-angle grain boundaries into a direct crystal plasticity finite element (CPFEM) simulation (see Fig. 1). Closely related to this topic are the questions of (i) which grain boundaries are susceptible to nucleate damage (cracks or voids) upon (strongly localized) plastic deformation and (ii) how to capture such susceptibility as function of strain within the framework of (direct) crystal plasticity. In order to gain further insight into those, numerical simulations performed on the compute cluster of the AICES complement the experimental efforts at the MPIE.

Dr. Varnik’s research activity at the AICES is focused on the applications of the lattice Boltzmann method (LBM) and extensions thereof on those types of problems which lie at the interface between physics



**Fig. 1:** Stress distribution (color coded) resulting from a direct crystal plasticity finite element simulation of a spherical indentation into an oligocrystalline patch of about 20 columnar grains (solid shading with thick lines indicating grain boundaries).



**Fig. 2:** An illustration of the lattice Boltzmann method (LBM) study of droplet dynamics on topographically patterned substrates.

and engineering science (see Fig. 2). In this context, a deep understanding of boundary conditions within mesoscopic approaches such as the LBM plays an important role. Under the supervision of Dr. F. Varnik, this problem is been addressed by Nima Hamidi-Siboni, one of the youngest members of the AICES within the doctoral programme. Despite the quite short time passed since his arrival on March 2008, Mr. Hamidi-Siboni has already been able to perform multiscale analysis of the finite volume approaches within the LBM, thereby shedding light onto the nature of the so called numerical viscosity appearing in some of these schemes [2].

Dr. Friák's proposed AICES projects are focused on (i) novel multi-descriptive approaches to the mechanics of polycrystals and (ii) quantum-mechanical study of the stress-enhanced stability of phases. The first is aimed at a methodological development

of new multi-scale approaches effectively combining both discrete- and continuum-formulated concepts, i.e., the extraction of decisive material parameters from the electronic structure density-functional-theory (DFT) calculations and an up-scale transfer of these characteristics into CPFEM simulations. Particular material systems in view are (i) biocompatible Ti-based alloys for biomedical applications and (ii) advanced Mg-based materials.

The second AICES project is focused on precipitates that are formed at grain boundaries where they become stable only due to the presence of strong local strains or stresses. As the formation of precipitates represents an important hardening mechanism in materials at medium and elevated temperatures, their stress-enhanced stability is of critical importance. Both projects are continuations of on-going inter-departmental (MU-CM) research activities [3,4] at MPIE.

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## Computational Mechanics of Polycrystals - CMC<sup>n</sup>

### A Joint Research Group of Max Planck Society and Fraunhofer Society



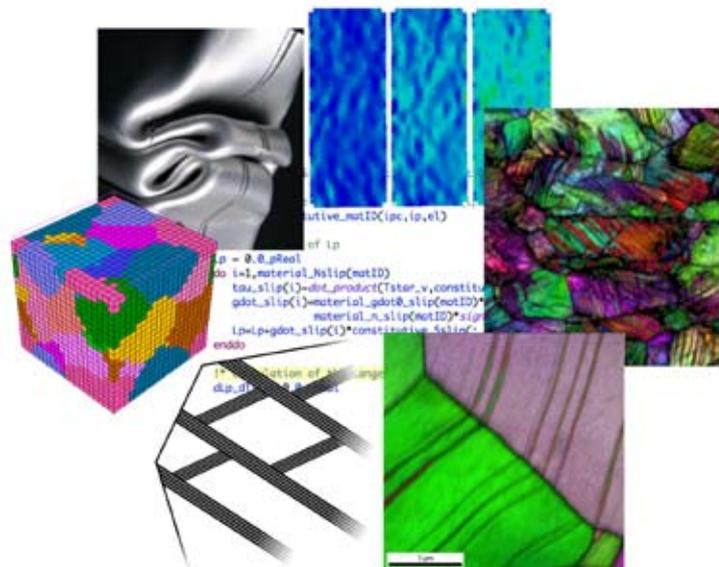
*P. Eisenlohr, L. Hantcherli, F. Roters, D. Raabe*

Most technically relevant metals are of polycrystalline nature, that is they are made up of a huge number of crystallites with sizes ranging from submicrometers to several millimeters. Since single crystals possess anisotropic mechanical properties, any non-random orientation distribution of the crystallites (so called crystallographic texture) usually results in anisotropic properties also for the polycrystalline material. During the recent past significant progress has been seen in both the physical understanding and the continuum mechanical simulation of polycrystals. The research in this field is characterized by a close connection between fundamental metallurgical insights and technological applications. Therefore, the Max-Planck-Gesellschaft, as Germany's most important organization for fundamental research, and the Fraunhofer Gesellschaft, as its application-oriented counterpart, in 2005 decided to establish a joint working group on "Computational Mechanics of Polycrystals – CMC<sup>n</sup>" starting in spring 2006. The goals of this initiative are set along two parallel lines: (i) a deeper metallurgical understanding of mechanical twinning, deformation induced phase transitions and multi-phase microstructures, and (ii) the optimization and acceleration of existing simulation methods both in Taylor-based and self-consistent texture modelling as in finite element method (FEM) based homogenization schemes.

Strategically, the CMC<sup>n</sup> initiative aims at a long-term alliance between the Max Planck Institut für Eisenforschung (MPIE, Düsseldorf) and the Fraunhofer Institut für Werkstoffmechanik (IWM, Freiburg) to accelerate the knowledge transfer from fundamental sciences to applications. Besides the collaborative research goals, CMC<sup>n</sup> therefore also aims to build up two tightly connected self-supporting working groups, one at each institute. Hence, the acquisition of third party funds is integral to the initiative. In accordance with the projected growth path of the joint initiative, both institutions acquired a number of grants to fund additional research. For the time being, the initiative is well ahead of the targeted

timeline for fund-raising. The proposal anticipated secured external funds of 0.65 Mio EUR while the overall budget of current externally-funded projects already totals 1.7 Mio EUR.

The research during the first two years mainly focused on the fundamental understanding of mechanical twinning and its consideration in texture models and crystal plasticity FEM (CPFEM). Moreover, an own code of a multi-grain Taylor-based homogenization scheme was developed. Further advances were made towards better numerical schemes and the discrete approximation of the crystallite orientation distribution function. Model predictions were compared to the texture development during tension tests on rolled TWIP (twinning induced plasticity) steel.



*Polycrystal mechanics research builds on the close integration of experimental investigations at different scales in order to understand the underlying deformation mechanisms and on advanced FE constitutive modelling efforts to cast these mechanisms and their interplay into the form of variational solvers.*

The current initiative contributes to improved polycrystal modelling on various scales. At the highest resolution, grain clusters are explicitly modeled using single crystal plasticity in each grain. The response to mechanical loading is usually simulated with the finite element method (FEM). On

this scale, the most important achievements in this work are an improved physically based twinning model for single crystal plasticity and the treatment of multiple phases in a representative volume element (RVE). Larger polycrystals subject to spatially homogeneous deformations are modeled with self-consistent and Taylor-based stand-alone texture models. Also here, the incorporation of an improved twinning model into the visco-plastic self-consistent (VPSC) scheme is a highlight of the performed work. Moreover, an own code for a fast Taylor-based grain interaction (GIA) scheme was developed and texture effects on load reversal were studied. The stand-alone texture models are also used in a combined homogenization scheme where medium sized grain clusters are considered at each integration point in a FEM simulation. Here, the most important progress was an extensive numerical study on the treatment of different phases within such a scheme. On the macroscopic scale, the polycrystalline nature of a

material is represented in anisotropic yield loci used in FEM simulations. On this scale, the development of a material model combining an established model for isotropic and kinematic hardening with an evolving anisotropy is the central achievement.

In response to the results achieved in the first two years, the members of the initiative secured funding from both MPG and FhG to continue the support for ongoing and new activities in the years 2009–2011.

The experience gained in this joint initiative during the first two research years has led to an adjustment of the project structure. The major change in comparison to the formerly favored layout of four projects is the broadening of software development into a unifying effort across the whole initiative, the streamlining of the original goals into three major projects plus the addition of demonstrations and industrial applications as shown in the following table.

<b>Scientific Board (Raabe, Gumbsch, Riedel, Roters)</b>			
<b>MPG-FhG initiative <i>Computational Mechanics of Polycrystals</i></b> ( two of the project leaders serve as coordinators: Dr. P. Eisenlohr (MPG), Dr. T. Hochrainer (FhG) )			
Project 1	Project 2	Project 3	Project 4
Physical mechanisms of crystal plasticity  L. Hantcherli	Homogenization  Dr. P. Eisenlohr	Models of texture evolution  Dr. T. Hochrainer	Demonstration and industrial applications  Dr. D. Helm
Fundamental aspects of deformation-induced phase transformations and mechanical twinning in TWIP and TRIP steels	Theory of homogenization and scalable Taylor models for polycrystals	Constitutive models for texture simulation and development of self-consistent texture models	Application of novel constitutive models and homogenization schemes to industrial examples
Software modularization and algorithms for continuum mechanical material point models			



## Christian Doppler Laboratory for Polymer / Metal Interfaces

G. Grundmeier, N. Fink, R. Posner, X. Wang, M. Valtiner

The Christian Doppler Laboratory for Polymer/Metal Interfaces funded by the Christian Doppler Society in Austria was established at the MPI für Eisenforschung (MPIE) in April 2003. Prof. Dr. Guido Grundmeier is leading this laboratory, which cooperates with voestalpine Stahl Linz and Henkel Austria as industrial partners. In July 2006 Dr. Grundmeier received his habilitation and *venia legendi* on “Interface Chemistry and Adhesion” from the faculty of mechanical engineering at the University of Bochum. In December 2006 he took over the position of a full professor and head of the Institute for Technical and Macromolecular Chemistry within the department of chemistry at the University of Paderborn. However, based on an agreement with the MPIE and the industrial partners, the laboratory was proceeded at the MPIE in Düsseldorf to guarantee the continuation of the work and the access to established sophisticated experimental techniques.

Currently, four scientific co-workers are working in the frame of three research modules at the MPIE.

While the focus in the first two years was on the understanding of conversion coatings [1,2], the focus was shifted to the fundamental understanding of adhesion and de-adhesion and the design of new interfaces and thin films. Furthermore, a fourth module was established at the University of Paderborn in October 2007 dealing with the mechanical and barrier properties of thin nanocomposite coatings.

After the third year of the CDL, the existing three modules at the MPIE were refocused on the following three topics:

**Module 1:** Understanding of the electronic and adhesive properties of passive films on Zn alloy coatings

**Module 2:** Correlation of microstructure of the substrate and the layer formation as well as the formability of thin hybrid conversion coatings

**Module 3:** Adhesion of polymer coatings on modified Zn alloy surfaces in environments of high water activity

Methods of electrochemical, spectroscopic and microscopic analysis were further developed to enable the characterisation of processes like degradation of interfaces or formation of defects in conversion layers due to forming of the substrates. Electrochemical set-ups were combined with other analytical tools like a miniature stretching apparatus (analysis of defect formation), an FTIR-ATR cell (analysis of water uptake and changes in interfacial composition) or the Raman apparatus (degradation of layers during oxygen reduction reaction) to get in situ information of changes in the layered system during the exposure. Complementary investigations were dedicated to the molecular understanding of adhesion based on the application of Chemical Force Microscopy and Single Molecule Adhesion Studies. These studies were combined with quantum mechanical simulations.

**Research module 1:** The main focus of this module was to understand electronic and adhesive properties of passive films on Zn-alloy coatings. As these protective layers are itself covered with a passivating ZnO layer, the focus was to first understand the adhesive behaviour of these passive films. For this purpose single-crystalline ZnO(0001) model surfaces have been prepared by an ambient atmospheres approach, featuring large atomically flat terraces as can be seen in [3]. The use of single crystalline surfaces allows well-defined experimental investigations of the ZnO surfaces which are com-

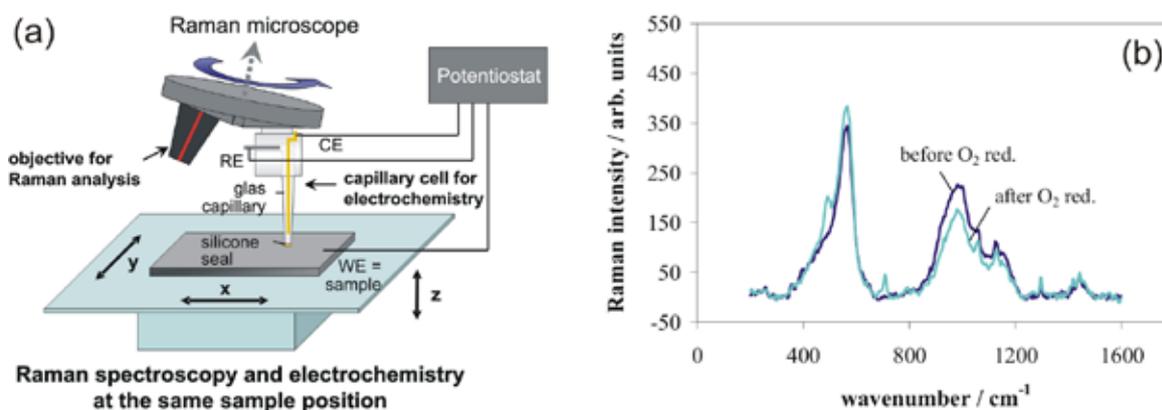
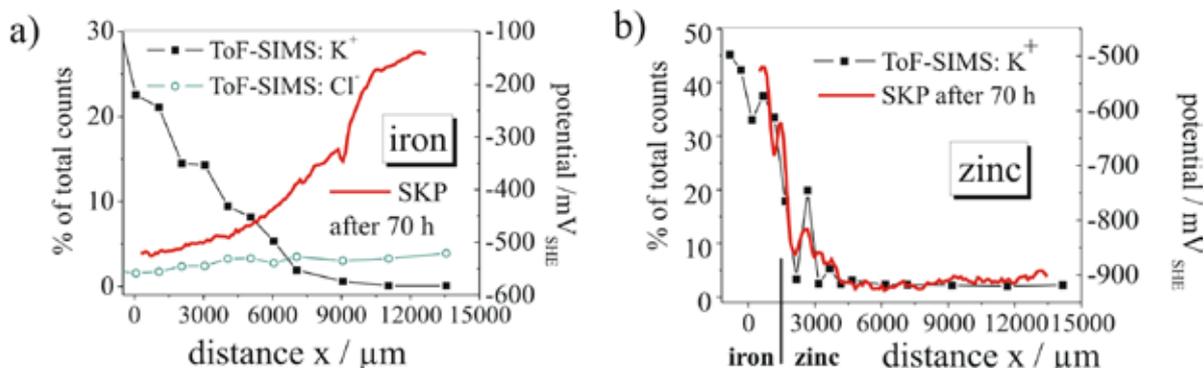


Fig. 1: a) schematic of the set-up for combined Raman – electrochemical measurements. b) Raman spectra of a conversion coating before and after the electrochemically induced oxygen reduction.



**Fig. 2:** Ion distribution on a polymer coated zinc/iron-sample after interfacial ion transport processes (starting from a 0.5 M KCl solution covered iron defect in humid nitrogen atmosphere). a) SKP potential profile of the iron area after 70 hours and corresponding ToF-SIMS line scan of the surface K<sup>+</sup> and Cl<sup>-</sup> distribution after coating removal. b) SKP potential profile of the zinc area after 70 hours and corresponding K<sup>+</sup> distribution.

parable to the constraints of ab-initio based simulations methods. This enables the possibility of a synergistic combination of experiment and theory. These ideal substrates were employed to understand the behaviour of ZnO in electrolyte environments at different pH levels. For high resolution in-situ AFM investigations a liquid-cell for force-distance and topography measurements under pH variation at non-equilibrium dissolution conditions was developed. This setup allowed the measurement of topographic changes of the ZnO surface during dissolution processes [4]. It could be shown that the prepared ZnO(0001)-Zn surfaces are stable and single crystalline in electrolyte environments over a wide pH range from 11 to 4. Thus these surfaces are an ideal substrate for adhesion studies within electrolyte environments. Furthermore, it was possible to reveal the dissolution mechanism of ZnO surfaces. The main important result was that the different crystallographic surface orientations feature totally different dissolution kinetics and mechanism. A detailed discussion of the dissolution mechanism is presented in the highlight report on p. 195.

**Research module 2:** With a new experimental set-up (see Fig. 1a) developed at the MPIE it is possible to perform Raman and electrochemical measurements in the same local area of a specimen. With this set-up it is possible to analyse the influence of oxygen reduction on conversion layers. This effect is important for a better understanding of the delamination behaviour (during delamination oxygen reduction takes place at the polymer/metal interface) of conversion layers which was analysed in former experiments [5]. Fig. 1b shows Raman spectra of a conversion layer before and after the electrochemically induced oxygen reduction (performed as potentiodynamic measurement in borate buffer with  $-100 \text{ mV} (E_{\text{OCP}}) < E < E_{\text{OCP}}, dE/dt = 1 \text{ mV/s}$ ). The intensity of the conversion chemistry signal ( $950\text{-}120 \text{ cm}^{-1}$ ) decreases due to the oxygen reduction while the ZnO signal ( $570 \text{ cm}^{-1}$ ) keeps almost constant in intensity after the electrochemical measurement. This means that a quite good protection of the substrate is still provided during the oxygen reduction process although the conversion coating itself already starts to degrade.

**Research module 3:** The established mechanistic model for ion transport processes at polymer/oxide/metal interfaces included ion diffusion as major driving force in humid nitrogen atmosphere. Diffusion was also regarded as important step to start and enable cathodic delamination on iron oxide and zinc oxide. Recent studies in contrast could prove that interfacial ion diffusion will not take place on these substrates on a macroscopic scale. A combined Scanning Kelvin Probe (SKP), X-Ray Photoelectron Spectroscopy (XPS) and Time-of-Flight Secondary Ion Mass Spectroscopy (ToF-SIMS) approach showed that solely cations of the defect electrolyte are transported along iron oxide at atmospheres of extremely reduced oxygen partial pressure (see Fig. 2), which disproves diffusion and confirms that positively charged ions are attracted by a negatively charged substrate surface. On zinc oxide no such process takes place (see Fig. 2), indicating that the charge density of the surface is at least extremely reduced. It was concluded that this charge should be an artefact of an initial hydroxide formation in air which will not be removed on iron oxide during deposition in nitrogen atmosphere. Additional experiments confirmed that the observed effects are independent from the coating type and structure. The mentioned studies led to a deeper understanding of the determining effects of ion transport at polymer/oxide/metal interfaces and will help to design interfaces with improved corrosion stability in the future.

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## Materials World Network: Physically Based Approach for Predicting and Minimizing Damage Nucleation in Metals

*P. Eisenlohr, Y.J. Ro, F. Roters, D. Raabe*

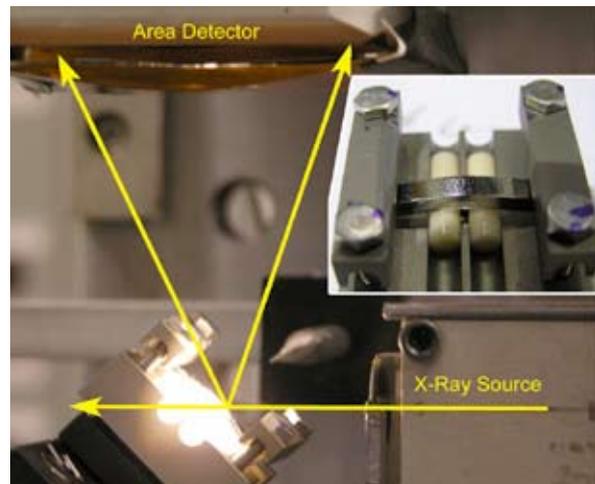
The ability to predict damage nucleation and evaluate whether it will lead to the fatal flaw is one of the major goals of computational plasticity. However, most modelling of damage is based upon the assumption of pre-existing flaws or cracks, and the modelling approaches developed so far predict the growth rather than the nucleation of damage. While heterogeneous deformation is understood to be a precursor to damage nucleation, the step between heterogeneous deformation and damage nucleation is not clearly understood. If heterogeneous deformation is not modeled accurately, then it is unlikely that damage nucleation and subsequent damage growth can be confidently predicted. From a review of current understanding of heterogeneous deformation and deformation transfer at grain boundaries, identification of mechanisms of damage nucleation will require quantitative knowledge of (i) the orientations of crystals on either side of the interface, (ii) the boundary orientation and structure (energy), (iii) the activated deformation systems on either side of the boundary, and (iv) the stress-strain gradient history in the grains on either side of an interface. We will investigate metals and alloys with simple microstructures that have an intrinsically low ductility (in this case titanium and dual phase steels) that will provide the best opportunity to gain the slip system based information needed to clearly identify damage nucleation mechanisms.

The goals of this research program are: (1) Identify fundamental rules for discriminating strong and weak boundaries in the context of a deformation path. (2) Express rules quantitatively in the form of models that track boundary strength as a function of local stress and strain history. (3) Implement grain boundary strength rules into computational models of mesoscale deformation. This will be accomplished in a joint research project involving Michigan State University (MSU) and Max-Planck-Institut für Eisenforschung (MPIE), where mutually useful skills are present which can reach the above goals when integrated into an international cooperative research program. The work will be carried out by three Ph.D. students under the guidance of Profs. Thomas Bieler and Martin Crimp at MSU, and Drs. Philip Eisenlohr and Franz Roters at MPIE.

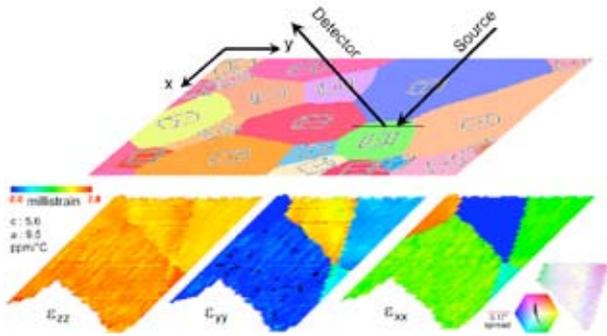
The research proposal has been granted individual funding from the US National Science Foundation (NSF) and the German Research Foundation (DFG, since mid-2007). At MSU, research is mainly carried out within the Ph.D. studies of Y. Yang and L. Wang. At MPIE, Dr. Yun Jo Ro started in July 2008 as a post-doc fellow in this research project.

The cooperative research is to be intensified by regular extended visits at each partner's institution. Up to now, two of such annual exchanges took place. Dr. Eisenlohr has spent about three weeks at MSU in May 2008. In return, a large delegation of guest researchers comprising Prof. T.R. Bieler, Prof. M. A. Crimp, Y. Yang, and L. Wang, complemented by Prof. D. Mason (Albion College, Albion, MI, USA) has visited MPIE for about five weeks in the summer of 2008. Both visits resulted in a strong boost of the research activity with each partner being able to fully utilize the facilities of the respective host.

Investigations started with rolled Ti of commercial purity having moderately strong basal texture of about 7 times random with basal poles in the transverse direction. Prior to four-point bending in a stage shown in Fig. 1, the spatially resolved crystal orientation is recorded on a significant portion of the overall sample surface by means of electron backscatter diffraction (EBSD, see top of Fig. 2 for example). Furthermore,

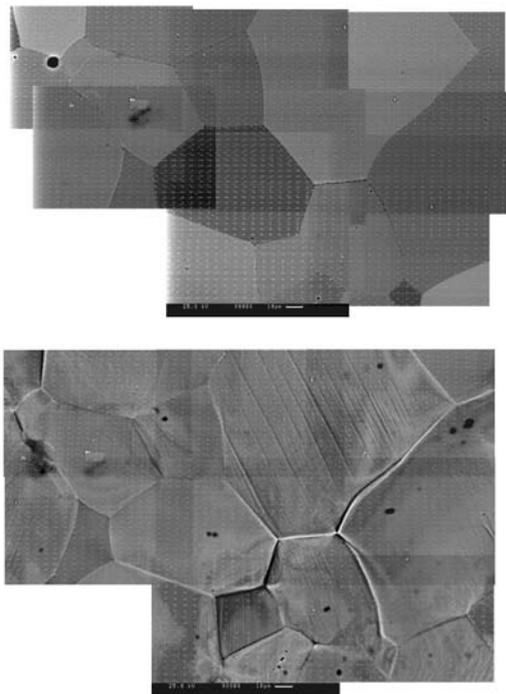


**Fig. 1:** Bent sample configuration and X-ray beams in the 3D X-ray microscope.



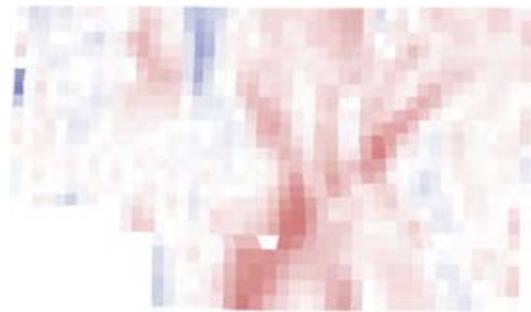
**Fig. 2:** Sub-surface grain morphology and internal strains present in undeformed sample; green grain on surface is highly misoriented from its left and right neighbors reflected by its strongly differing internal strain.

a grid of Pt-markers is applied in selected regions via focused ion beam (FIB)-assisted deposition. Optionally, a 3D X-ray facility at the Advanced Photon Source of Argonne National Laboratory allows for quantifying the elastic strains and crystal orientations in selected slices perpendicular to the surface (see Fig. 2).



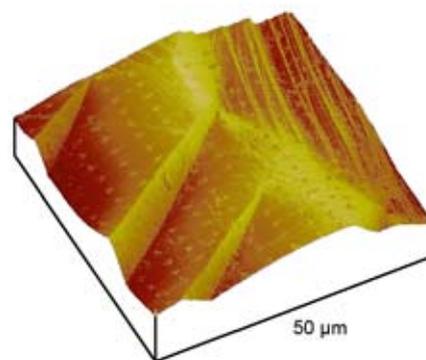
**Fig. 3:** Montage of back-scattered electron contrast images before (top) and after 8% horizontal tensile surface strain (bottom). Pt-markers form grid of light points. Linear features within strained grains correspond to slip traces.

After deformation, the samples are again characterized by means of EBSD. From the correlation of scanning electron microscopy (SEM, see Fig. 3) images the surface strain map in marked regions is accessible as illustrated in Fig. 4. The surface topography evolving during four point bending is quantified with the help of atomic force microscopy (AFM, Fig. 5) to get, e.g., the full displacement vector of mechanical twins that emerge in various grains



**Fig. 4:** Map of horizontal strain component from differential image correlation of Fig. 3. Scale runs from -0.3 (blue, compression) to 0.2 (red, tension).

and mediate plastic deformation. Electron channeling contrast imaging (ECCI) is able to reveal linear traces of either dislocation slip or mechanical twinning and helps in identifying the predominant plasticity mechanisms as function of grain orientation. In order to measure the grain boundary inclination and reveal sub-surface features of deformation, a FIB-milling is employed which is additionally capable of preparing samples for high-resolution transmission electron microscopy (TEM) observations.



**Fig. 5:** Atomic force microscopy image of grain boundary triple point showing dominant twinning in lower grain.



# New Scientific Laboratories and Facilities

## Christian Doppler Laboratory for Diffusion and Segregation Mechanisms during Production of High Strength Steel Sheet

M. Rohwerder

The new CD lab was founded on January 1<sup>st</sup> 2008. The main focus is on fundamental diffusion and segregation problems encountered during the different production steps of high strength steel sheet. Cooperation partners are voestalpine and TU Wien.

The technical motivation behind this is that the development of high strength steels with their characteristic alloying element composition leads to new challenges for the production and processing of steel sheets in order to meet the product requirements. Following this growing demands, it is no longer sufficient to produce steel with good materials properties. Future steel sheets not only have to be harder, to be easier to manufacture, to show excellent corrosion resistance, but in addition it also is important that they can be processed resulting in steel sheet with perfect surface condition. Newly developed high strength steels show unexpected and harmful surface effects. The technological impact of these effects is detrimental and presents an urgent task for the future. This project combines the applied research focusing on the technological requirements and basic research in an ideal manner.

These detrimental effects occurring during the pre- and post-processing of high strength steels sheets are fully based on the mobility and diffusion of metallic and non-metallic alloying elements. This mobility leads to enrichment processes on the surface or within the steel sheet structure thus influencing the quality of the final product. Among the non-metallic elements especially hydrogen and oxygen needs to be addressed, as these compounds are obviously adsorbing on the steel surface and are diffusing into the sheet during the necessary pre- and post-processing steps.

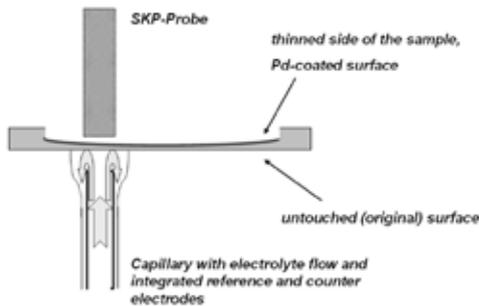
The related questions are of significant scientific interest and the related research of considerable experimental challenge. For instance, for these steel grades the measurement of the extremely low, but problematic, hydrogen uptake into the steel during cleaning, annealing and galvanizing is far from straight-forward.

In sum these processes are addressed within the lab in three modules: SE-, P- and H-Module.

**SE Module:** The phenomena observed during hot rolling of high strength steels such as surface



**Fig. 1:** A highly sensitive micro-balance (right hand side) was built up and integrated into a highly-flexible environment that enables automated and computer-controlled gas supply of well-controlled composition. Together with a high-speed IR-furnace set-up (built up for the RFCS project “Novanneal”) the microbalance shares a mass spectrometer (left hand side at the window) to which it is connected via a capillary.



**Fig. 2:** Electrolyte is flushed through a small capillary onto the sample surface. Directly opposite to this the Kelvin Probe is positioned at the exit side. When the measurement is finished after a few minutes the Kelvin Probe is moved to the next position and soon after that (after the signal has stabilised) also the capillary is moved (to the position under the Kelvin Probe). In such a way many measurements on the uptake kinetics during cleaning, pickling and electro-galvanizing can be carried out on one and the same sample.

oxidation, grain boundary oxidation and segregation are unexceptionally based on diffusion correlated enrichment processes in related activity gradients. Especially the presence of air during hot rolling leads to adsorption and dissociation of oxygen on the sheet surface and its diffusion into the alloy along grain boundaries. The correlated grain boundary oxidation and segregation are at the focus of this module. A process related analysis on the one hand and a basic understanding of the specific role of alloying elements on the other hand is necessary in order to develop counter measures to avoid the severe problem of enrichment processes. The thermodynamic and kinetic modelling of these processes shall provide a basis for future predictions of enrichment phenomena, as a basis for steel developments. These processes are described in the module 1: 'Selective enrichment in hot and cold rolled strip' (SE-Module).

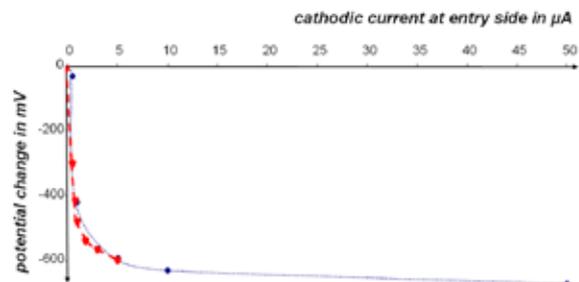
**P-Module** (main activity at TU Wien, Prof. Danninger): The micro-topology of the pickled hot rolled strip surface is detrimental for the further workability in the following production routes. The major challenge in pickling high strength steel sheets is a consequence of the enrichment processes of reactive elements like phosphorus and silicon as described above, leading to a grain boundary dominated surface structure with a completely changed micro-topology. With increasing pickling time, deeper parts without grain boundary oxidation are pickled, thus leading to a change in the surface topology with negative consequences for further processing routes (i.e. change of emissivity upon annealing). As different pickling times cannot be avoided in the production line, the surface properties along the strip axes are unforeseeable thus creating big problems in the following aggregates. A possible solution for this problem can be the implementation of pickling inhibitors in order to suppress the dissolution of the steel substrate. The pickling behaviour of low alloy steels, selection, efficacy and technological questions of pickling inhibition are task of module 2:

'Pickling and pickling inhibition of low alloy steels' (P-Module).

**H-Module:** The hydrogen uptake of the material is a significant problem especially for high strength steels, as small amounts of hydrogen in the range of ppm can cause retarded brittle fracture. As the sensitivity of this phenomenon is given by the structure and composition of the steel itself, the only solution is to influence the kinetics of hydrogen uptake into the alloy during the different critical processing steps. For that, it is of particular importance to investigate the mechanisms of hydrogen uptake during the relevant processes i.e. pickling, electrolytic cleaning, acid dip, galvanic zinc-coating and recrystallisation annealing in order to develop measures for a retarded uptake. These mainly electrochemical investigations are task of module 3: 'Hydrogen uptake' (H-module).

Since January 2008 the main activity within the lab was to built up the required experimental infrastructure required for the planned investigations.

This includes a microbalance for studying in-situ the kinetics of grain boundary oxidation (see Fig. 1) and a novel experimental set-up for in-situ monitoring of hydrogen uptake, one based on the Kelvin Probe technique (see Fig. 2) and one based on permeation into an ultra-high-vacuum chamber and subsequent detection by mass spectroscopy. Although this lab is quite new and the experimental environment had to be built up first, there is one main result that shall already be mentioned here: the high sensitivity of the Kelvin Probe technique for sensing hydrogen uptake into metals. This is based on the fact that the potential at the exit side of the metal (covered by Pd) will be pulled down from its relatively high potential that corresponds to its high work function towards the potential of the hydrogen electrode. According to the Nernst equation the dependence is a logarithmic one, emphasizing the small concentrations (see Fig. 3).



**Fig. 3:** Potential change measured by Kelvin probe on the exit side as a function of cathodic current at entry side (about  $0.504 \text{ cm}^2$ ). The Pd membrane was  $25 \text{ }\mu\text{m}$  thin. The blue curve was obtained with the electrolyte purged by nitrogen, the red one by argon. The slight deviations of the curves at low currents are due to traces of oxygen in the nitrogen purged electrolyte. The red curves demonstrates how sensitive the method is: about  $300 \text{ mV}$  at  $-0.5 \text{ }\mu\text{A}$  current, about  $420 \text{ mV}$  at  $-0.75 \text{ }\mu\text{A}$ , about  $480 \text{ mV}$  at  $-1 \text{ }\mu\text{A}$ . Above  $-1 \text{ }\mu\text{A}$  the method becomes increasingly insensitive. At  $-2 \text{ }\mu\text{A}$  about  $540 \text{ mV}$  are measured and at  $-5 \text{ }\mu\text{A}$   $600 \text{ mV}$ . We are confident to be able to detect hydrogen uptake as low as  $50 \text{ nA}$  or even lower than that.



## Novel Scanning Kelvin Probe for Integration into Synchrotron Beam Line

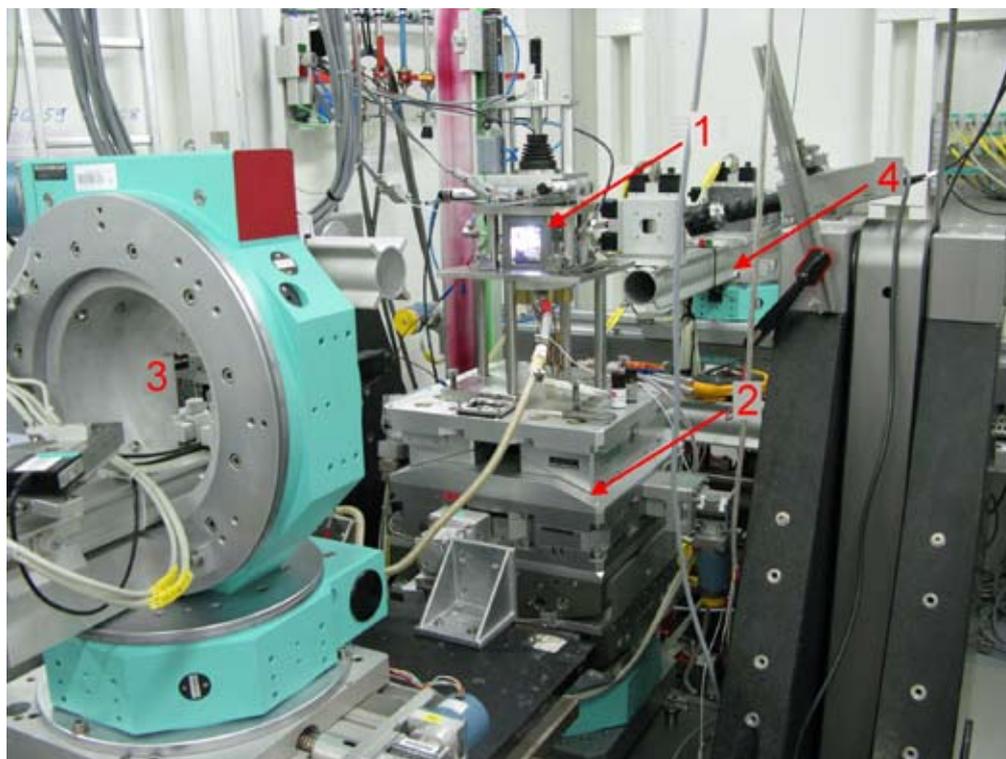
M. Rohwerder, D. Vogel, B. Salgin

The great advantage of a Scanning Kelvin Probe (SKP) is that it enables the in-situ measurement of work function (or dipole potential) changes at the buried interface of many layered systems. In cooperation with Dr. Reichert (dept. Prof. Dosch) of the MPI für Metallforschung in Stuttgart we made use of this for the study of two systems: ice/silicon and ionic liquids on sapphire. Investigations by synchrotron radiation diffraction suggested dipole moments at the respective buried interfaces that gave rise to controversial discussion. Hence, these systems were preliminarily investigated at MPIE by SKP, confirming this to be an ideal complimenting technique. Even more interestingly, as SKP provides localized information about dipole changes, SKP was recognized as an ideal tool for investigating in

how far the synchrotron beam might affect structures observed with it: by scanning across the sample interface hit by the beam and outside the beam can be directly measured. If the SKP signal measured over the beam differs from the one left and right of it, then this would mean that the beam indeed affects the interfacial structure, because this would directly be due to an affected interfacial dipole moment.

Hence, an SKP set-up was built up at MPIE that can be integrated into synchrotron beam lines (see figure of the SKP installed in the HEMD set-up at ESRF, in cooperation with Prof. Dosch, MPIM).

The first results obtained indicate that indeed the beam may affect the interfacial structure. However, further research is required for substantiating this.



*Photography of the SKP installed in the HEMD set-up: The High Energy Micro Diffraction (HEMD) setup is a permanent setup, installed at the backend of the ID15A beamline of the ESRF. The SKP (1) was assembled on a heavy-duty sample tower (2) for high accuracy positioning of the sample, which is assembled on a massive granite base. The tilt monochromator (3), which was not used for this experiment and the detector tower (4) are designed as completely independent assemblies.*

## Optical Floating Zone Furnace

*A.W. Hassel, S. Milenkovic*

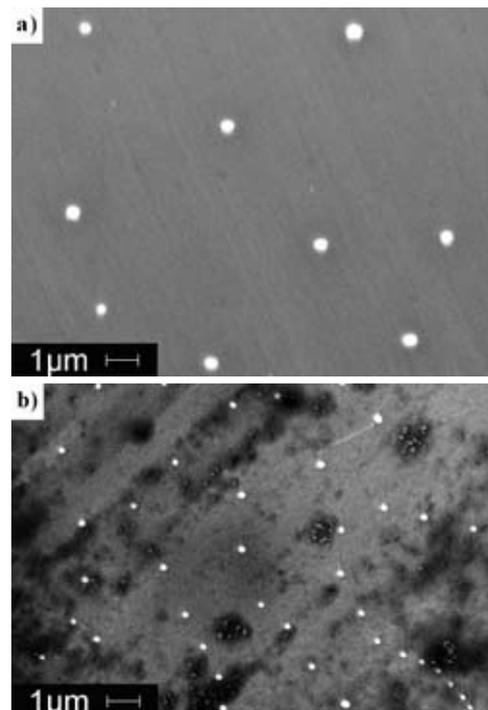
The floating zone technique is a powerful tool for the fabrication of high quality single crystals and for the purification of materials. A new four mirror optical floating zone furnace FZ-T-800-H-PC (Crystal System Co.) was installed, that consists of four ellipsoidal mirrors made of Pyrex glass coated with highly reflective aluminium. The mirrors are air-cooled in order to prevent moisture condensation on the ellipsoidal surfaces. The principle of this technique is that radiation from the Halogen lamps is reflected and focused by the mirrors onto the bar sample to form a molten zone at the tip of the feed rod. Then the molten (floating) zone is translated along the sample length by moving the mirror stage with respect to the sample. The crystal is grown on the solidifying end of the floating zone. In addition, a rotation movement of the rod improves the microstructural homogeneity during directional solidification. The oven is custom made and is in particular equipped with magnetic sealings to allow an operation under inert gas or in high vacuum. Translation and rotation of seed and feed rod can be independently set to allow better mixing and diameter modulations.

A very successful research direction in nanotechnology has been established during the last years in the Electrochemistry and Corrosion group that employs directional solidification of eutectics for the preparation of nanowire arrays. For this application the floating zone method has several advantages. Firstly, it is a crucible free method which prevents contamination of the sample from a

reaction with crucible. Further, the starting material can be in the form of sintered powder samples, rather than cast pre-alloys. Next, as only the small zone between feed rod and seed rod is molten, the time for gravitational concentration differences is shorter. Especially eutectics or monotectics of a light and a heavy element, such as Al-Bi, which tend to show gravitational segregation, can be stabilized by this approach. Moreover, this method enables a more sophisticated investigation approach in which discs of different composition are sintered as a stack. During the subsequent zone melting of the sample the feed rod always feeds a composition different from the composition into the molten zone, resulting in a continuous change of the composition. This allows determining the correct composition for systems in which the phase diagram is not sufficiently known and/or gradient samples for combinatorial alloy development. Finally, the relatively small amount of material necessary for this type of sample production is one more advantage, thus allowing to process platinum metal eutectics (Ru, Rh, Pd, Os, Ir, Pt), for the production of their nanowires, an approach which was up to now restricted by the high costs for preparing the required amount of noble metal pre-alloy. The last important advantage is the higher temperature gradient which allows producing much smaller nanowires as it was already confirmed in the NiAl-W system in which the smallest W nanowires had a diameter of ca. 150 nm from the Bridgman method and ca. 75 nm with the Floating Zone Method.



*Optical floating zone furnace*



*NiAl-W eutectic directionally solidified in a) Bridgman furnace and b) Optical furnace*



## Transmission Electron Microscopy Laboratory

A. Kostka

A Jeol JEM-2200FS Field Emission Transmission Electron Microscope operating at 200kV is the most powerful instrument recently installed in our laboratory. It is equipped with an in-column energy filter, a Gatan CCD camera, a Jeol EDX system, three scanning TEM (STEM) detectors (an upper high angle annular dark field (U-HAADF) detector, a bright field (BF) detector, and an HAADF detector) and a piezo-controlled goniometer (see figure).

Features of the JEM-2200FS include a high-brightness Schottky field emission electron gun producing a probe size of less than 0.2nm. The ultra-high point-to-point TEM resolution is 0.23nm; atomic scale resolution of 0.1nm can be achieved. The facilities are ideally suited for crystallographic and chemical analyses at a sub-nanometer scale, including high-sensitivity EDS and EELS. Both EDS and EELS are analytical TEM (ATEM) techniques and can provide elemental composition and distribution information. The Gatan high resolution CCD sensor allows capturing highly-detailed EELS and energy-

filtered TEM (EFTEM) data sets with maximum throughput. The HAADF-STEM imaging is an important tool for the study of materials by forming HAADF (Z-contrast) images that are sensitive to chemical composition variations of the sample. The microscope is mainly used for TEM, STEM, EDS and in future EELS studies of a wide spectrum of materials.

Most of the specimens are prepared using a Jeol JEM-9320 focused ion beam system which provides simple, fast and precise sample preparation. This generation of the FIB is equipped with a side-entry goniometer stage for TEM specimens which allows for direct TEM investigations of the prepared specimen without transferring it into another TEM holder. The advantage of such a solution becomes very clear when the TEM inspection reveals that the specimen must be further thinned. The software records the last processing position and the milling can be easily continued until optimum thickness is achieved.



*Transmission electron microscope Jeol JEM-2200FS. All operations are performed using a remote control system unit which is located in the next room.*

## X-Ray Diffraction Laboratory

H. Pinto

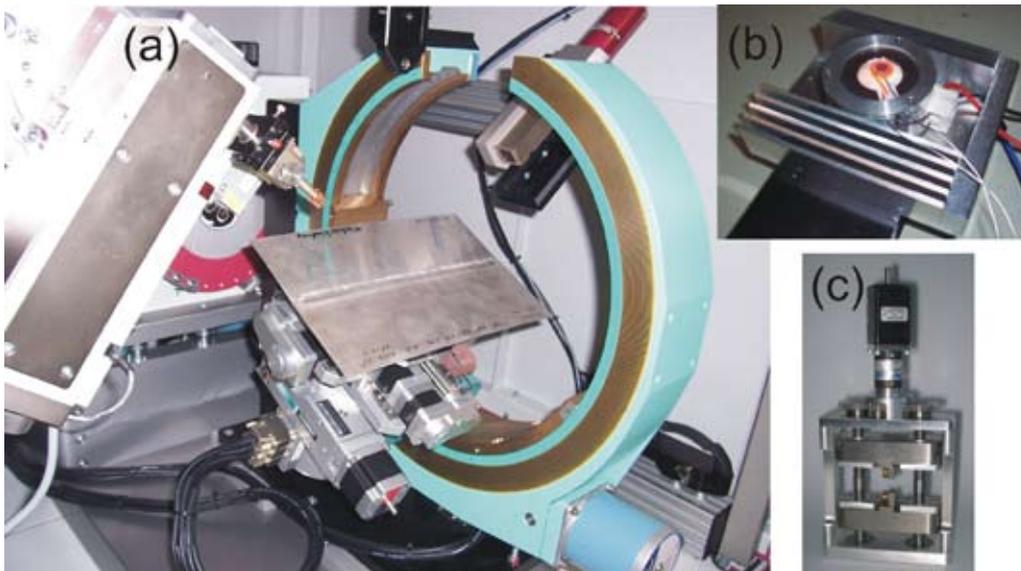
In the past decades diffraction methods employing characteristic radiations emitted from X-ray tubes became a powerful technique for studying crystalline materials. The limited penetration depth of lab X-rays into hard matter results in an attractive surface sensitivity which usually suits to the analysis of near-surface regions and thin films. Owing to the interaction of X-rays with the atomic arrangements and the element specificity of the scattering power, X-ray diffraction (XRD) allows for determining within the diffracting volume average values of microstructure properties such as phase composition, preferential grain orientations, internal stresses and defect densities as a function of manufacturing and/or service parameters. XRD is furthermore non-destructive, being nowadays an indispensable tool for materials engineering in research as well as industrial sectors.

The MPIE invested in 2006 in two new 4-circle diffractometers ( $\theta$ - $2\theta$  and  $\theta$ - $\theta$  geometries) designed by GE Inspection Technologies which enable phase and stress analyses of more flexible sample shapes and sizes (see figure) as well as the use of additional environments during in-situ investigations. Within the last 2 years an air-cooled resistance furnace for oxidation in air at temperatures up to 1100°C as well as a miniaturized tensile testing device for uniaxial deformation was developed by the group Materials Testing to fit onto the diffractometer

stages. In addition, both equipments operate with a parallel-beam setup consisting of a polycapillary semilens in the primary beam and a 0.15° Soller slit in the diffracted beam. This setup minimizes in both equipments angular displacements caused by slight sample misalignments. Grazing-incidence diffraction (GID) can also be performed to study thin films.

The already existing X-ray facilities at MPIE which suited exclusively to powder measurements have been upgraded within the report period to more versatile setups. Nowadays the laboratory offers three further diffractometers: a 4-circle goniometer for texture measurements, a 2-circle diffractometer equipped with a graphite monochromator and a 0.15° Soller slit for stress analyses of massive samples using the  $\omega$ -modus and for phase analyses and a diffractometer for in-situ high-temperature investigations under controlled atmosphere equipped with a high-temperature chamber and a 120° curved position sensitive detector.

The operational control of the diffractometers was also upgraded to LINUX platforms running the well-established Scientific Software for diffraction purposes SPEC which controls nowadays several synchrotron X-ray and neutron diffractometers worldwide. SPEC provides a reliable and flexible instrument control for accurate sample alignment, single crystal as well as powder diffraction experiments.



(a) 4-circle  $\theta$ - $\theta$  diffractometer for phase, texture and stress studies; (b) air-cooled resistance furnace for in-situ oxidation experiments; (c) miniaturized tensile testing machine for in-situ uniaxial straining.



## Spark Spectroscopy for Materials Processing

S. Weber

The laboratory for metallurgy at the MPIE is not new, but several improvements and investments were done within the report period. Generally, a wide range of advanced facilities is operated to produce and refine high melting materials, most of them steels, with capacities up to 80 kg and temperatures up to 1900°C. A significant improvement was introduced end of 2008 by the commissioning of a new spark spectrometer, primarily for the chemical analysis of materials during the processing.

The poor reproducibility of castings done in the metallurgy of the MPIE was criticized for a long time, not only by external project partners but also from within the institute. A process control was only possible for determining the contents of carbon, nitrogen and hydrogen in steels, while a full analysis could not be realized. To overcome this restriction and establish a full analysis during the process, at least of iron-base alloys, the institute invested in an optical emission spectrometer in 2008. A state-of-the-art system on the basis of semiconductor detectors was delivered by the company OBLF (Witten, Germany),

allowing the parallel analysis of 25 alloying elements in iron, including exceptional elements like Hafnium and Rhenium. A full list of elements and limits of quantification is given in Table 1. The spectrometer features a Paschen-Runge line-up temperature stabilized to +5°C allowing the measurement of the full wavelength range from 130 to 680nm. The semiconductor detectors used were specially developed for spark spectroscopy and guarantee the same sensitivity as found in conventional spectrometers equipped with photomultipliers. The system was delivered and installed in September 2008 and is now used for the process control of three of the four available vacuum induction furnaces. For this purpose, a separate laboratory for sample preparation, measurement and storage of retain samples was equipped.



Spark Emission Spectrometer VeOS

Element	Limit of Quantification [wt%]
B	0,0005 - 0,5
C	0,003 - 3,0
N	0,002 - 1,0
Al	0,002 - 6,0
Si	0,002 - 4,0
P	0,001 - 0,5
S	0,001 - 0,2
Ti	0,001 - 2,0
V	0,001 - 5,0
Cr	0,005 - 30,0
Mn	0,005 - 30,0
Fe	Matrix Element
Co	0,005 - 10,0
Ni	0,005 - 40,0
Cu	0,005 - 3,0
Nb	0,003 - 0,5
Mo	0,003 - 10,0
W	0,005 - 10,0
Ta	0,005 - 1,0
Y	0,003 - 0,2
Hf	0,003 - 0,2
Zr	0,003 - 0,2
La	0,003 - 0,2
Ce	0,003 - 0,2
Re	0,003 - 0,2
Nd	0,003 - 0,2

List of elements and limits of quantification of the VeOS spectrometer



## Thermodynamic Scientific Laboratory

J. Garcia

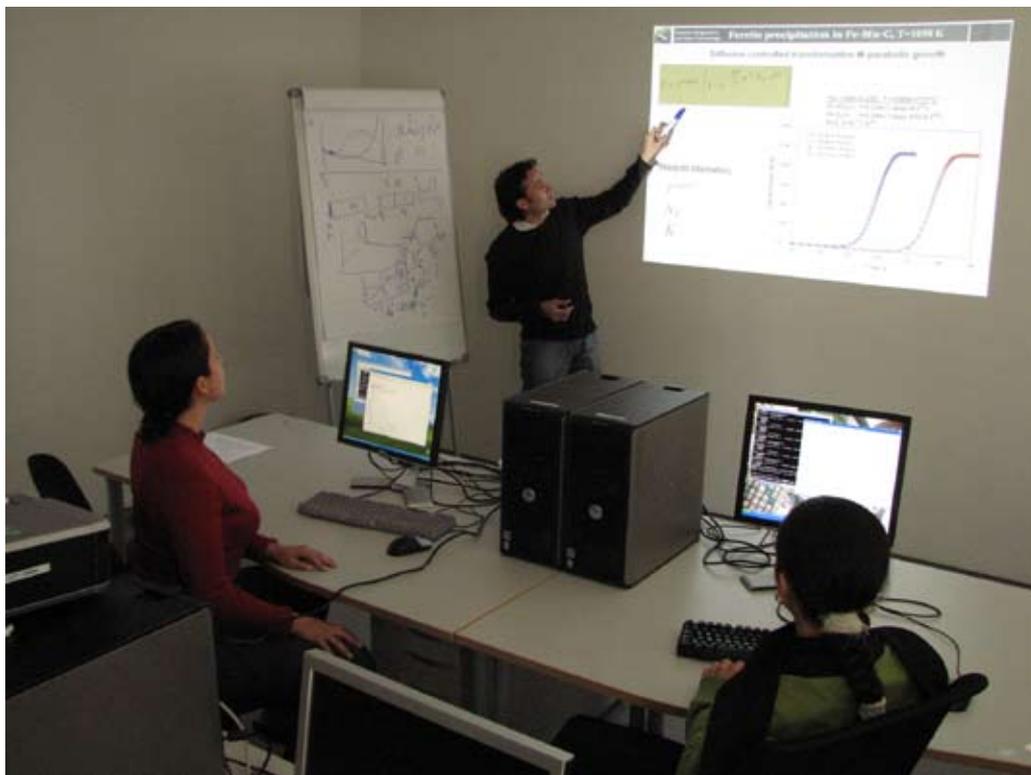
A new scientific laboratory for “Thermocalc and Dictra Calculations” was created by Dr. Garcia and Dr. Weber including a room equipped with a full computer facility for an *interactive learning method* at the department Werkstoffdiagnostik und Technologie der Stähle.

The thermodynamic scientific laboratory offers regularly trainings and seminars in the “Thermodynamic seminar room” covering topics from basic lessons on principles of thermodynamic of alloys and diffusion-controlled processes, as well as the implementation of computational thermodynamics for the development of alloys and/or processes.

The basic courses, which include a basic training in the use and application of the software, are carried out by Dr. Garcia and Dr. Weber. Case studies and problems to be solved by the scientific staff can be discussed and assessed interactively.

For advanced courses and applications of ThermoCalc and Dictra, the group is supported by the expertise of Prof. Dr. Gerhard Inden, especially on the field of kinetics of phase transformations in multi-component systems.

Invited seminars carried out by international experts are foreseen.



“Thermodynamic seminar room”



## CM Computer Centre

S. Boeck, W. Vogt, U. Wellms

The computationally demanding simulations which are performed in the CM department as well as the atomic simulation group of the GO department require a powerful and scalable high-performance computing (HPC) center. To keep pace with changing simulation trends a flexible concept has been developed which unites flexible administration of manifold HPC and network architectures, a user-friendly environment that supports efficiency and cooperation for the users, reliability of the provided services, and last but not least security measures.

Thanks to the new building of the CM department the computer center was provided with new rooms to build up a computer center that can meet the demands for the performed simulations. The newly available rooms of 60 m<sup>2</sup> allow to expand the center with the needed compute power. Cooling capacity and electric power supply of 200 kW are now available. The water-cooling technology applied in the initial stage has proven to be very reliable and cost-efficient. In contrast to conventionally cooled computer centers only the volume inside of the racks is air-conditioned, i.e., 20 m<sup>3</sup> instead of 180 m<sup>3</sup> air volume. Besides lower investment costs the high efficiency of the water cooling system saves approx. at least 10% energy per year compared to conventional cooling methods. Besides the 4 racks from the initial stage we implemented 5 additional water-cooling racks in the basement of the new building. The setup of 7 additional racks is already prepared. In January 2009, the currently on-going european tender procedure will boost the compute power from 120 nodes (320 CPU cores) to 320 nodes (1920 CPU cores). Since the performed simulations require MPI parallelization the new cluster will be equipped with a fast interconnect to perform massive-parallel simulations. The total performance of the new compute center is estimated to be 20 TFlop/s ( $R_{max}$ ) and a total storage capacity as large as 60 TB (20TB storage with backup, 40 TB scratch space).

Based on the experiences obtained during the initial stage three years ago, a new HPC compute center could be constructed from scratch: (i) The successful object-oriented administration approach based on the scVenus framework has been fine-tuned, (ii) the multi-platform and multi-OS concept has been implemented and (iii) the *single points of failures* could be identified and replaced with a robust nearly high-available (nearly HA) concept. The latter one has been developed in cooperation with Science&Computing and Audi. The successful unattended installation method has now been extended to work also with the vital master nodes. The new ansatz is based on a double RAID Level 6 NetApp file server.

We further optimized the integration of all PCs of the department into the cluster system. All nodes and PCs are virtually mapped to a huge single

(heterogenous) system. Users can freely choose between SuSE Linux und Windows XP Professional (64bit) as their PC's host system while accessing the counterpart by XWin32 or rDesktop. All file spaces (home, raid, san, scratch) are mapped transparently. Our single sign-in solution avoids tedious daily user jobs like file transfer between clients with different operating systems.

An optimized monitoring system helps reducing the impact of possible hardware failures to users. All vital parameters of the compute nodes, the PCs, all file servers, as well as the components of the cooling systems (pumps, valves) are constantly monitored (24x7 every 2 minutes). Minor difficulties can be identified before a system actually crashes. Critical problems (failure of disks, shelves, or power supplies) trigger automatically an order of spare components via email ("self-healing").

The unattended installation of PCs and compute nodes allow the user instantly to continue their work on a spare PC while the broken machine can be repaired. The actual down-time can be reduced to a minimum. With a general up-time of 99.7% over the past 2 years and operation periods of 450 days and more users can perform simulations without interruptions for months.

Our concepts combines a fine-tuning of hard- and software to achieve peak computational performance, keep down-times small, and offer a flexible and user friendly system to the users.



*New computer center of the CM department consisting of 200 high-performance compute nodes, 20 TFlops (estim.) with scalable state-of-the-art water cooling system (up to 200 kW capacity).*

## Unified Network Information System

R. McCormack

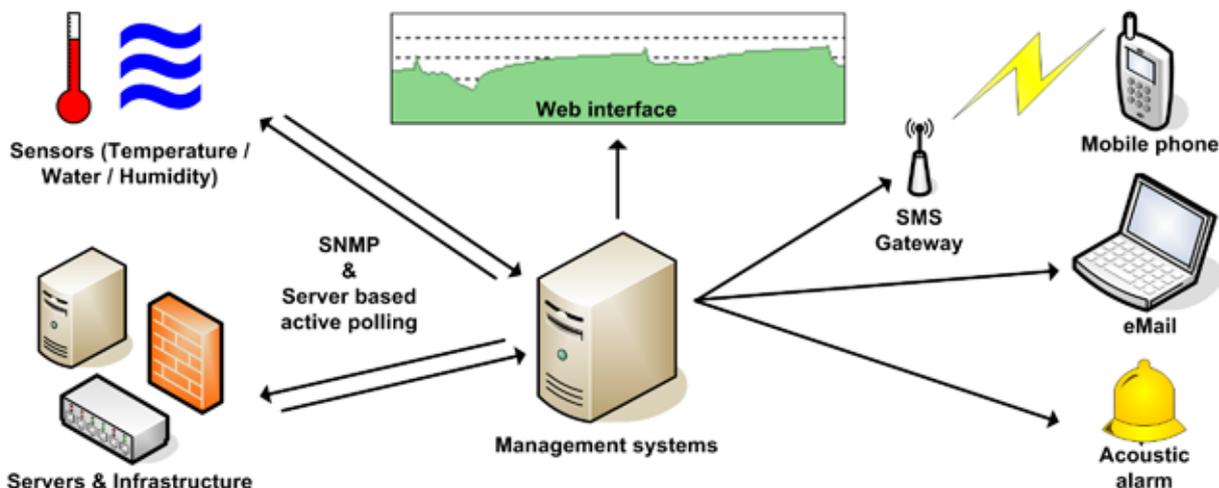
The Network Centre was established in 2005 and has since successfully completed numerous large IT projects, which are accessible by all departments. The Ethernet network has seen continuous growth and now consists of approximately 2000 Fast Ethernet Ports and 500 Gigabit Ports spanning across multiple buildings at our Max Planck Institute. In recent times, a large Storage Area Network, with multiple servers offering around 50 Terabyte of storage for all departments, was implemented. A multi tiered, high redundancy firewall strategy from different vendors was integrated seamlessly into the network. This greatly reduces the risk of a similar security flaw in different products, allowing all users ample 100 Mbit Internet access. Multiple existing and also new server systems have successfully been virtualised allowing not just fast recovery but also better use of the server hardware. In the first phase of the virtualisation project, 18 servers have been virtualised saving approximately 7,5 kW/h of combined power and cooling costs.

To keep our “virtual eye” on a large number of systems, network hardware, servers and environmental values such as humidity and temperature, the Network Centre has invested a great deal of effort to build a highly redundant and scalable monitoring and alarm system. The Netmanagement, as we call it, consists of multiple stages of active and passive monitoring. Computer systems can signal a problem via active SNMP traps and custom monitoring agents, or in case of a complete failure, the central

Netmanagement system can register the event. Vital rooms and systems are also monitored by independent network agents, which constantly check the temperature and monitor for water breaches in these locations, in case of which, they can notify the centralised system of the problem.

Since a monitoring system is worthless if it can't convey its message, the system was designed to notify the Network Centre staff via a custom audio-visual system, emails and/or SMS messages. To really make this system powerful, it was crafted so that all the systems have access to the whole scope of the alarm notification system, making it not only highly redundant but enabling a very large scope of incidents to be reported in multiple ways. If for instance an air conditioning system in a server room fails, the rising temperature is registered, a Short Text Message is sent to the mobile phones enabling the technical staff to take appropriate action. The Network Centre also gets informed and can monitor the temperature via a web based bar graph allowing access to this information from virtually anywhere.

After a trial period, the Network Centre offered the use of this equipment to all departments as a “low cost, high gain” technology to monitor the ambient temperature of sensitive equipment such as Tunnel Electron Microscopes, or in case of a fatal failure of the system, directly notifying a service technician via SMS of the failure and thus directly helps to significantly reduce the downtime of a highly valuable piece of scientific equipment.



Schematic diagram of the alarm system



## PART II.

# THE DEPARTMENTS

(in alphabetical order)

<b>Department of Computational Materials Design</b> <i>(J. Neugebauer)</i>	<b>45</b>
<b>Department of Interface Chemistry and Surface Engineering</b> <i>(M. Stratmann)</i>	<b>61</b>
<b>Department of Material Diagnostics and Steel Technology</b> <i>(A.R. Pyszalla)</i>	<b>81</b>
<b>Department of Materials Technology</b> <i>(G. Frommeyer)</i>	<b>95</b>
<b>Department of Microstructure Physics and Metal Forming</b> <i>(D. Raabe)</i>	<b>105</b>





# Department of Computational Materials Design

J. Neugebauer

## Scientific Concept

The aim of the department Computational Materials Design is to develop and apply highly accurate simulation tools for the prediction of materials properties and processing, which are feasible on present day computers. The main challenge is the inherent multiscale character of realistic materials: While from an engineering point of view the processes on the macroscale determine the suitability of a material for industrial applications, the fundamental scale which eventually controls these properties is the electronic scale. At this scale the relevant particles are individual atoms and their interaction is described by quantum mechanical laws which govern the formation, stretching, bending, or breaking of bonds. In realistic engineering materials the two scales are connected via a highly complex microstructure which extends over several orders of magnitude in length and time scale (see Fig. 1).

While traditionally in simulating engineering materials single scale or top-down approaches have been employed, recent advances in quantum-mechanical methods and in computer performance opened the unique opportunity to turn this approach upside down and to start hierarchical simulations on the most fundamental level. This bottom-up approach has exceptional features: Since it is based on the well

established laws of quantum mechanics, it is free of any adjustable or empirical parameters (*ab initio*), the elementary building blocks such as electrons and nuclei used in the simulations are identical to those in nature, and all equations defining the dynamics of these building blocks are well known. Therefore, these *ab initio* based multiscale simulation methods provide the unique possibility to design and predict novel materials solely on the computer and promise thus faster development cycles at lower cost.

A practical realization of such a bottom-up approach for engineering materials faces a number of challenges. Key issues which have been investigated by the department and for which major methodological developments have been performed are listed in the following:

- For realistic systems a full quantum mechanical solution is numerically not feasible and approximations have to be used. The method of choice in materials science is density functional theory (DFT), which relies on the accuracy of the underlying exchange-correlation functional. The evaluation of the performance of available functionals in predicting materials properties is crucial, since all errors made at this most

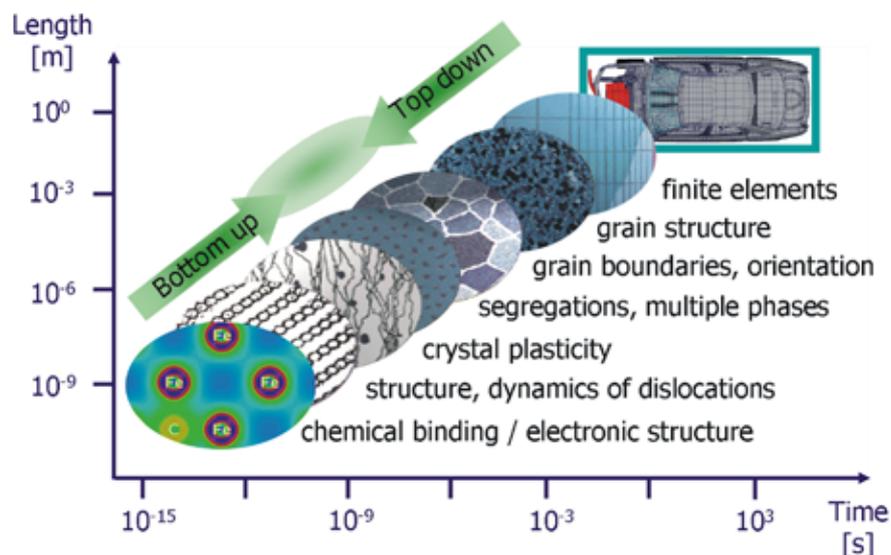


Fig.1: Multi-scale approach in materials design

fundamental level are propagated to the largest scale [1]. Further, the development of simulation techniques beyond conventional DFT such as the exact exchange formalism (EXX) [2-4] or quasiparticle formalism [5-7] is essential.

- Even on high-performance computer clusters, as the one at the MPIE, system sizes which can be treated by DFT are limited to a few hundred atoms and a few thousand configurations. Using conventional expressions to transfer information from the smaller to the larger scale typically results into a complexity which is numerically not feasible. For an illustration consider the anharmonic contribution to the free energy: Using a direct approach as e.g. molecular dynamics and requiring a numerical/statistical accuracy of  $\sim 0.1$  meV, up to  $10^6 \dots 10^7$  configurations have to be considered. Thus, the development of efficient coarse graining techniques cannot be restricted to the length and time scale but has to be extended also to the configuration space.
- A key quantity to determine static and dynamic materials properties is the free energy. For engineering materials a major challenge is the large number of excitation mechanisms (such as harmonic/anharmonic vibrations, electronic/magnetic excitations, chemical disorder, point defects and their interactions) which all contribute [1,8,9]. Therefore, multiscale approaches have not only to bridge between different scales but also have to combine areas in physics/ chemistry which so far had not been connected (multi-physics simulations).
- A full bottom-up approach with a complete transfer and coarse graining of all parameters is often not feasible even on state-of-the-art supercomputers. Therefore, a crucial aspect is the construction of new multiscale approaches which are more approximate but can be applied already on present-day computers to solve open materials science problems [10].

To address this wide range of aspects the department has been structured in five scientific groups which each work on one or several of these topics in multiscale development. The group of S. Boeck (Algorithm Design and Modelling) has worked on a modular and highly flexible framework to implement and test multiscale algorithms (S/PHI/nX). The group of T. Hickel (Phase Studies) has developed highly advanced approaches and sampling techniques to compute the various free energy contributions such as harmonic and anharmonic vibronic contributions, electronic and magnetic excitations, point defects with a numerical/statistical accuracy of better 1 meV. The group of M. Friák (*ab initio* Thermodynamics) investigated various homogenization methods which allow an effective scale jumping from *ab*

*initio* computed properties such as phase stability and elastic properties to macroscopic behaviour of polycrystals or complex biological structures. Finally, the group of L. Lymperakis (Microstructure) developed length-scale bridging techniques to study the thermodynamics and kinetics of extended defects such as dislocations and grain boundaries. To extend the activities also to surfaces and to non-metallic systems such as oxides and to supplement the recent experimental spectroscopic activities in the Department "Interface Chemistry and Surface Technology" a new group "Defect chemistry and spectroscopy" has been launched in Sept. 2007 and is headed by C. Freysoldt. This group is also closely linked to algorithmic developments in the S/PHI/nX multiscale library.

In the past two years each of these scientific groups made tremendous progress in their respective fields. In addition to the own developments the department has acquired expertise in numerous state-of-the-art simulation tools such as e.g. cluster expansion and the generation of quasi-random structures using ATAT, large-scale molecular dynamics calculations using LAMMPS and IMD, or spin and classical Monte Carlo using ALPS. Using this machinery of high level multiscale and more approximate scale-jumping techniques the department has been able to cover a wide variety of materials science questions ranging from very fundamental topics in materials science to topics of high relevance to industry. Recent achievements and highlights will be discussed in more detail in the reports of the respective groups and in the selected papers and cover a wide range of materials. The main focus has been on ferritic and austenitic metallic alloys [1,8-13] but materials studied include also advanced semiconductors [14,15] needed, e.g., for optoelectronic emitters or photovoltaic elements, shape change [16] or shape memory materials [17], corrosion at surfaces, or even biological systems [18] and chitin-based nanocomposites.

While initially many of the studies and projects have been performed by a single group more and more activities use the combined expertise and spread over two or more groups. This joint expertise enabled to address critical materials science questions having a complexity which exceeds a realistic treatment by single methods. A prominent example in this respect is the study of hydrogen embrittlement which is a major potential failure mechanism in modern high strength steels. To design alloys which are less sensitive to this failure mechanism it is crucial to understand the underlying mechanisms. Since these span over several length and time scales several group expertises had to be combined to successfully address these questions: Calculation of solubility energies and diffusion barriers in bulk alloys including magnetic effects, of binding energies and diffusion



Fig. 2: Participants at the international workshop on *Ab initio* description of Iron and Steel (ADIS2008) organized by the CM department

barriers in and along selected grain boundaries, or the effect of H on dislocation-dislocation interaction (see e.g. p. 209). Other examples of inter-group and often even inter-departmental activities are the development of *ab initio* based strategies to identify new classes of light-weight alloys (p. 139), the study of TWIP/TRIP steels or of biological nanocomposites (p. 169).

Based on this joint expertise the department also has been highly successful in building up joint research projects within the MPIE with the departments of Prof. Raabe (see e.g. selected papers p. 139 and p. 169), Prof. Stratmann (p. 195) and Prof. Frommeyer (p. 131). These strong interdepartmental activities helped to establish and participate in major regional scientific networks such as the Interdisciplinary Center of Advanced Materials Simulation (ICAMS, p. 17) or the collaborative research centre “Stahl-*ab initio*” together with the RWTH-Aachen (p. 19). It also enabled the department to be highly successful in initiating a broad range of scientific projects financed by external funding agencies or industry. Examples are several BMBF (ministry of education and science) projects with topics on high-temperature resistant steels, photovoltaics or energy efficient high-brightness white light emitting diodes, DFG (German Research Council) projects on the nucleation and initial microstructure formation in eutectic steels in collaboration with the RWTH Aachen and the IWF in Dresden, on materials for energy efficient optoelectronic emitters, or the Max-Planck Initiative on Multiscale Materials Modelling of Condensed Matter (Triple-M, p. 21) which provides a platform for joint methodological developments within the Max Planck society. The strong focus and experience of the department on multiscale approaches related to advanced materials questions make it also attractive for collaborations and longer visits from experts

specialized on various electronic structure aspects, i.e., on aspects which are on the most fundamental level of *ab initio* based multiscale simulations. For example, Dr. Mark Rayson received a prestigious award from the Alexander von Humboldt foundation which allows him to work for one year in the CM department. His study on a new generation of electronic structure algorithms for large systems, which in contrast to many previous approaches work equally well for semiconductor and metallic systems, opened a number of interesting applications.

The department has also organized and co-organized several international conferences and symposia, which were able to attract the top experts in the field (p. 253). A particular highlight was the international workshop on *Ab Initio* Description of Iron and Steel (ADIS2008) in June 2008 at Ringberg Castle. It was focused on the description and the various effects of magnetism in steels and related alloys. Like the first ADIS-meeting in 2006, it provided a scientifically very fruitful atmosphere of exchange and new ideas and following the unison wish of the participants will be continued as a biannual conference series. Another highlight was the joint interdisciplinary symposium at the Spring Meeting of the DPG Condensed Matter Section on “Modern developments in multiphysics materials simulations”.

Although the department started its activity only in summer 2005 its scientific contributions have been internationally widely recognized and members of the department have received numerous invited talks at key international conferences. Also, despite the main field of the department – multiscale simulations of engineering materials – being still in an early stage the opportunities opened by this connection of most fundamental physical concepts such as quantum mechanics and modern steels were



**Fig. 3:** In May 2008 the CM department moved into a completely reconstructed building.

not only recognized early on by industry but also spurred the imagination of non-experts: A number of interviews and press reports covering the work and vision of the department were published in major newspapers and popular science journals. A broad public outreach had also the participation at major science expositions like the exhibition “Max-Planck

– Revolutionär wider Willen” in Berlin or the IdeenPark of the Thyssen-Krupp initiative “Zukunft Technik entdecken” in Stuttgart where the CM-department had together with ICAMS a booth with interactive computer demonstrations on multiscale modelling and its impact on understanding/designing advanced alloys.

A great event and improvement in the department’s everyday work conditions was the completion of the reconstruction of the CM-building (formerly “Blauer Bau”) in May 2008. Before, the department members had been scattered for some years all over the institute, the employment of new members was severely limited by office constraints and a further extension of the computer cluster was not possible.

Moving into the new CM building which had been reconstructed from scratch and which provides now space for a large high-performance computer cluster consisting of up to 12 racks and with a maximum cooling capacity of 200 kW, a state-of-the-art conference room and well equipped offices gave a further great boost to the entire department and allowed for much more intense interactions between the department members.

## Scientific Groups

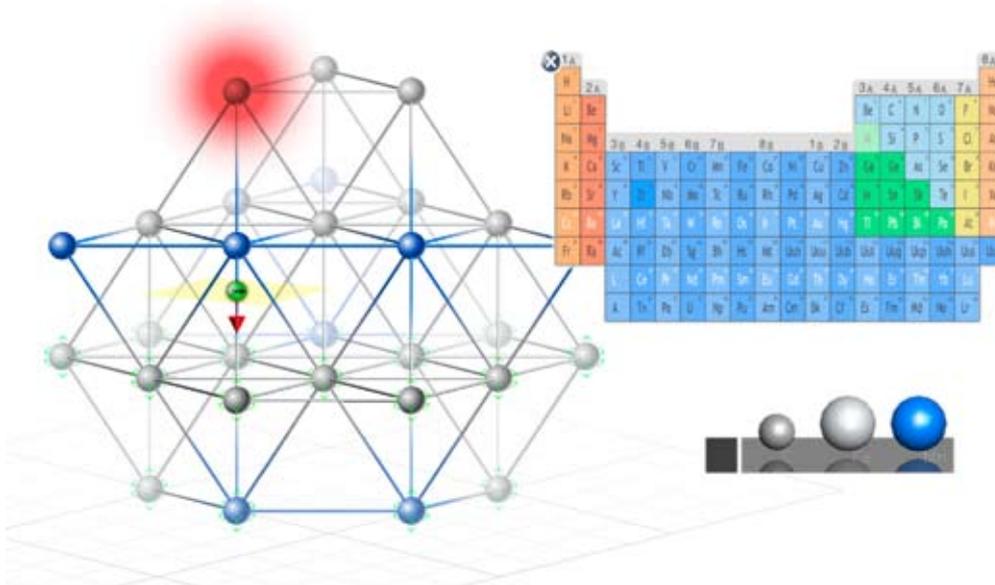
### Algorithm Design and Modelling (S. Boeck)

The steadily growing demand for an accurate description of material properties requires a thorough understanding of the underlying effects on various length and time scales, in particular, in predicting electronic and atomic structures, describing kinetics and thermodynamics, constructing structural-property relations, characterizing microstructures as well as building up material databases. In these disciplines there is a constant need of improving and developing efficient physical and numerical algorithms with respect to accuracy and performance.

**Ab initio multiscale library S/PHI/nX.** The Algorithm Design and Modelling group focuses on providing new methods and tools to simplify and speed up the process of method development in these fields. With the S/PHI/nX library we introduced and maintain a modular C++ framework for developing new numerical and physical algorithms. Currently, the library has been optimized for describing the electronic structure efficiently. A new programming technique (template linkage) has been developed that allows the introduction of the quantum mechanical language directly in the source code. Mathematical

expressions and Dirac projections are automatically transformed into efficient BLAS routines. It makes it easy for beginners to understand, modify, and test parts of the code while keeping peak performance.

**Scientific Database Concepts.** With more efficient codes and growing computer power the scientists in the CM department could perform far more simulations with multiple methods / codes on multiple scales. Particularly, when investigating trends of material classes the scientists often find themselves confronted with a flood of data, with respect to both size and amount of data sets. In the past 12 months the CM department submitted approx. 100.000 jobs to its cluster. Based on this number in average a Ph.D. student submits and analyses 20.000 jobs during a 3 years study time. The multi-scale nature of the research performed in the CM department requires using various codes with different input and output formats and units. With this amount of data it becomes hard to keep track of the generated data and to put them into a correct context. Besides the problem of organizing the data when investigating material trends an exchange of data becomes necessary. Therefore, a solution which can automatically keep track of performed simulations,



**Fig. 4:** Screenshot of an interactive PHInaX session by means of cubic  $\text{Fe}_{24}\text{Mn}_8\text{C}$  fcc supercell to model Mn-rich austenitic steels. In order to provide an interactive editor, graphic gizmos have been developed such as an automatic collision detector gizmo (red halo), multiple plane selector, guide points to place atoms/lattice points accurately. All gizmos have full access to the S/PHI/nX library and can facilitate symmetries and, in future, interactive atomic structure relaxations.

a sophisticated search engine, and a groupware solution is required for the on-going projects. Within the PHInaX project these issues are being tackled. In S/PHI/nX, the typical job preparation and analysis tools can be easily developed as S/PHI/nX add-ons. Add-ons are typically small (less than 50 code lines) and have a standardized input/output format and modifications can easily be implemented. PHInaX is able to map automatically the input and output of S/PHI/nX add-ons to databases without giving up performance or security issues. [19] Hence, the developer of a S/PHI/nX add-on implicitly updates the database structure. The on-going method development in the department causes frequent changes of input and output. The data cannot be stored by simply extending the database columns. Our approach stores relations between physical units (S/PHI/nX add-ons). This way every S/PHI/nX developer updates the database without any knowledge requirements on database interactions or database design. This approach makes sure that the stored information is being kept minimal which results in a high performance.

Since PHInaX stores physical data of different simulation codes in a unified unit system and keeps track of the physical content, this storage model gives rise to various new possibilities: (i) classification of data set reliabilities: A major problem in databases (such as material databases) is the determination of the reliability of the data. In order to make sure whether the data can be used for trend analysis the context of how the data set was obtained is crucial. Usually, this information is not available. In our approach the physical context of all data

remains accessible. (ii) Cluster usage: Sometimes similar calculations are performed from different users. Within the new approach a database tool can check if similar data can be obtained already from the database prior to job submission and computer resources can be saved. The new data base concept has been a subject of a diploma thesis (T. Uchdorf) done at the CM department and successfully defended at the FH Jülich [19].

**Scientific visualization: PHInaX.** While the database approach simplifies managing the increasing number of data, the Algorithm Design and Modelling group also concentrates on analysing the data sets themselves. The increasing compute power and memory sizes of modern computer architectures generate complex and huge data. In order to comprehend them an efficient visualization is required. When developing new physical and numerical algorithms pure visualization often does not suffice anymore. We therefore develop an interactive visualization module [20] and will integrate that into PHInaX. In contrast to conventional visualization software, PHInaX has full access to all physical algorithms implemented in S/PHI/nX and can incorporate them directly into the graphic render pipeline. This method allows new ways of interactive visualization: S/PHI/nX provides an efficient symmetry operation detection which can be visualized by PHInaX to simplify the building process of atomic structures. S/PHI/nX's fast structure relaxations using, e.g., EAMs can be incorporated into the editor to perform on-the-fly relaxations while constructing the atomic structure. The latter approach allows also performing theoretical "in-

*situ* experiments” before considering expensive *first-principles* investigations. It also allows to use future code developments in S/PHI/nX directly for interactive visualization (V. Bubnik) or to create input directly from the database.

Based on these features an intuitive human-computer interface (HCI) for *interactive* visualization of materials-design related data sets has been developed. For example, it allowed a successful implementation of a new atomic structure editor which provides the construction of even complex atomic structures within minutes. Currently we develop an *interactive* interface for scalar and multi-dimensional vector field visualization. The render engine has been designed to be compatible with new technologies such as virtual reality input devices and multi-touch displays. Based on the S/PHI/nX library the new approach allows direct incorporation of physical algorithms directly into the render pipeline to support interactions based on actual physical algorithms. The new HCI has been developed in the scope of a Master degree thesis, defended at the University Brno (awarded “Best Thesis of the Computer Science department”).

With PHInaX a new tool will be available which will help the scientists to keep an overview over their work, simplifies information exchange, automatically puts data into their physical context, and provides an module to visualize and in particular, interact with the data.

### Computational Phase Studies (T. Hickel)

The research in the group “Computational Phase Studies” is devoted to the physics of (meta)stable thermodynamic phases in metals and transitions between them. A major aim is the prediction of phase diagrams, due to the direct relation to many technologically relevant properties and processes in metals. In the last couple of years the research activities in the group have been focused on the development and application of state-of-the-art *ab initio* techniques employing density functional theory (DFT) in the following fields:

- The substantial improvement of the accuracy and efficiency in determining all relevant free energy contributions, since this provides the basis for an error-controlled prediction of thermodynamic quantities and phase diagrams.
- The description of first and second order (pre)martensitic phase transitions in a magnetic shape memory alloy, since this system serves as a model system to understand the general mechanisms behind this kind of transitions.
- The consideration of impurities and extended defects, in particular in high-strength steels, since they can have strong influences also on thermodynamic stabilities.

All these aspects are strongly interlinked and contribute to the general aim of the group. The calculation of accurate free energies is at the heart of many materials science problems and therefore the basis for intensive collaborations with several groups within the MPIE and beyond.

The *ab initio* calculation of free energies has been a central issue of the group, already since its foundation in September 2005. The main challenge is related to the fact that DFT is originally designed to predict ground state properties only. Its extension to finite temperatures means that excitation processes carefully need to be taken into consideration. The first research activities in the group have proven that the quasiharmonic approximation is an excellent method to describe the entropy contribution due to lattice vibrations over a large temperature range [21]. Taking additionally electronic excitations into account, a remarkable agreement with data obtained from Thermo-Calc has been obtained for pure non-magnetic metals [1].

However, noticeable deviations between the theoretically derived and experimental data have been observed close to critical temperatures related to magnetic, structural and solid/liquid phase transition, and hence in temperature ranges being of particular importance for phase diagrams. A major aim of the group has been therefore to improve the methods such that numerical accuracies systematically below 1 meV can be achieved. Accordingly, one research activity (F. Körmann) has been devoted to an accurate simulation of magnetic entropy contributions with

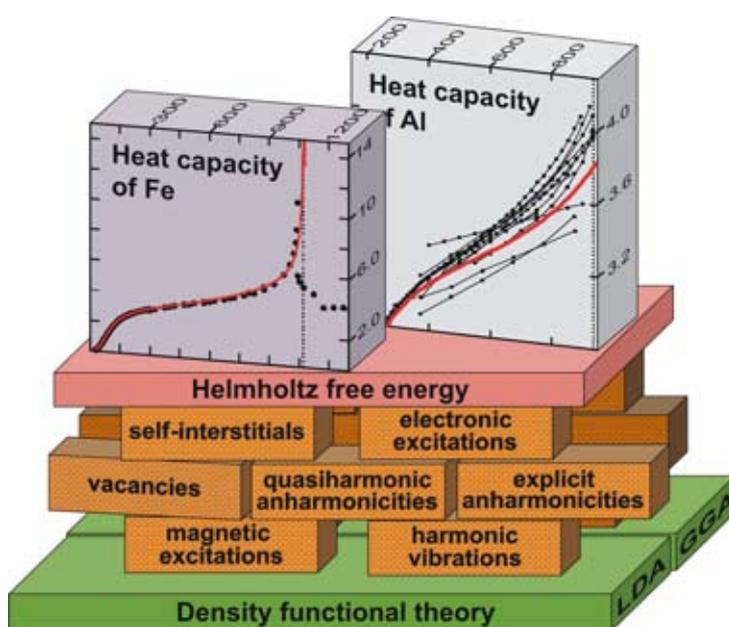


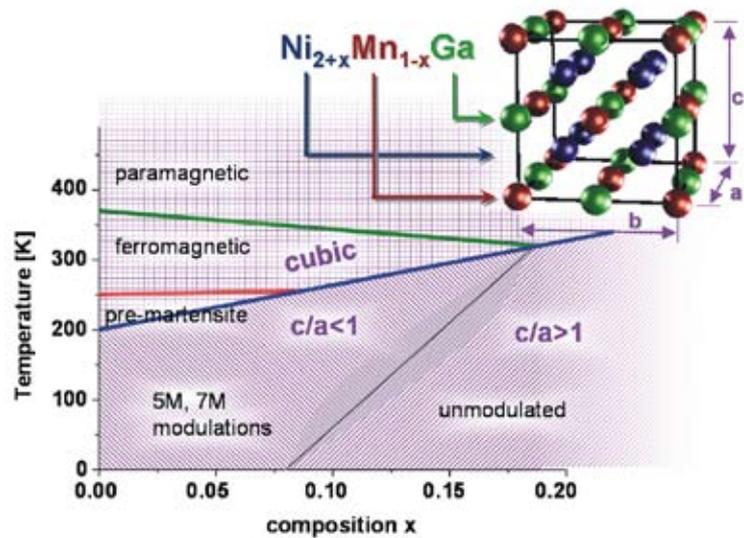
Fig. 5: Comparison of experimental (dots) and *ab initio* derived (red lines) heat capacities (in  $k_B$ ) of Fe and Al as a function of temperature (in K). The results are based on highly accurate methods to determine all relevant contributions to the Helmholtz free energy. The LDA/GGA approximation is used for Al/Fe, respectively.



a main emphasize to correctly reproduce the singular behaviour around the Curie temperature. Since classical approaches showed significant shortcomings in determining magnetic free energies, we had to develop techniques which allow for a proper quantum-mechanical treatment and which are based on *ab initio* data [8,11]. These techniques have been successfully applied to bcc iron (Fig. 5).

Another research activity (B. Grubowski) has been initiated by the observation that the quasiharmonic approximation shows small but significant deviations from experiment close to the melting point indicating that explicit anharmonicity (i.e. going beyond the quasiharmonic approximation) and/or entropic contributions due to the presence of point defects such as vacancies or interstitials [22] have to be included. Calculating these contributions turned out to be a major challenge since a numerical accuracy in all these contributions below 1 meV was crucial. Using sophisticated multiscale techniques to coarse-grain configuration space, the group succeeded in achieving this extreme high precision. Employing this approach for Al it became possible to reproduce the experimentally observed behaviour in the heat capacity close to the melting temperature (Fig. 5). This hitherto not achievable accuracy allowed e.g. to resolve a long standing debate which started already in the 60s whether vacancies or anharmonicity dominate the free energy close to the melting temperature. The availability of these methods initiated a number of projects and discussions within the MPIE, with the RWTH Aachen and TU München (Prof. W. Petry). For more details on this work, see p. 205.

**Magnetic shape memory alloys** have recently attracted a lot of excitement, since they allow shape changes of more than 10% with a frequency in the kHz regime - a property which is known to be related to a martensitic phase transition. The material system  $Ni_{2+x}Mn_{1-x}Ga$  is the most promising candidate for applications, but its operation temperatures and ductility still need to be improved. For this purpose an extension of the currently very limited knowledge on the phase diagram (Fig. 6) and the nature of the transitions is decisive. Based on the above mentioned techniques, these issues have been addressed (M. Uijtewaal). For the first time it became possible to describe the phase transition in detail, to reveal the involved delicate interplay of vibrational and magnetic excitations and to accurately predict the transition temperature [16]. A detailed analysis has explored the nature of the observed soft phonon modes and allowed the identification of phases, serving as a



**Fig. 6:** Phase diagram of the shape memory alloy  $Ni_{2+x}Mn_{1-x}Ga$  (Courtesy of K.-U. Neumann). The material is due to the ferromagnetic ordering (green line) and lattice deformations of more than 10% during the martensitic transition (blue line) very attractive for applications. *Ab initio* investigations help to resolve the controversy about the nature of the pre-martensitic (red line) and various intramartensitic (grey) transitions.

precursor effect to the martensitic transition [23]. These research activities are embedded in a priority program of the DFG.

**In high-strength steels**, such as Fe-Mn alloys the stacking fault energy is a decisive indicator for the probability of a martensitic phase transition. Therefore, the development of methods for an *ab initio* determination of its value and temperature dependence is a central issue of the group (A. Dick). In comparison to the other materials an additional challenge of Fe-Mn alloys is the huge configuration space of possible atomic and magnetic structures. These difficulties have been tackled by combining and evaluating a large set of advanced methods, including the concept of  $\gamma$ -surfaces, ANNNI models as well as cluster expansion and quasi-random structures [9]. The calculations for a wide range of Mn contents have revealed a hitherto unknown sensitivity of the stacking fault energy to the composition and the lattice expansion. These results will allow a detailed understanding, why certain steels predominantly show the TRIP or TWIP effect, and a meaningful prediction of promising material compositions. The consequences are explored within the collaborative research center "Steel - *ab initio*" and are also discussed with the MU department. For more details on this work, see p. 213.

The embrittlement experimentally observed in high-strength steels is closely related to another lattice defect investigated in the group, namely the incorporation of interstitial hydrogen. When studying a large set of metals [24], a remarkable dependence of the solution enthalpy on the lattice constants and the electronic configurations of the host atoms has

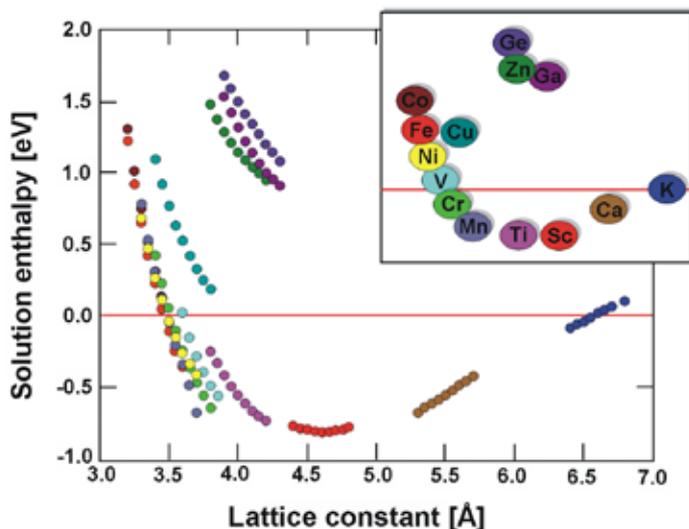


Fig. 7: *Ab initio* determined solution enthalpy of hydrogen in various metals (colour coded). The plot clearly indicates a relation of the solution enthalpy and characteristic lattice constants of the metals.

been detected (Fig. 7). The knowledge could be used for purposefully influencing the hydrogen solubility in a materials design. Since realistic steels are not single crystals but contain high concentrations of point and extended defects a detailed analysis of the interaction of hydrogen with vacancies, self-interstitials and grain-boundaries has been started. This study will allow a realistic description of thermodynamic properties such as solubility or hydride formation and kinetic behaviour of H in such steels (L. Ismer, U. Aydin).

Surface corrosion due to the presence of a thin electrolyte water film on the surface of steels is another technologically highly relevant issue. Based on the CM department's expertise on defect chemistry in semiconductors a new approach has been developed (M. Todorova), which provides a direct and intuitive link between *ab initio* accessible quantities and quantities relevant for the electrochemical reactions studied, e.g., in the GO department (Prof. Stratmann). This approach has been successfully applied to derive Pourbaix diagrams, which provide a fundamental concept to understand the stability of surfaces with respect to electrochemical reactions, see p. 199. Another result of this collaboration were combined experimental/theoretical studies of oxide surfaces, which lead to a detailed insight into surface structures and the mechanisms causing them, see p. 195.

### **Ab initio Thermodynamics (M. Friák)**

The group is primarily focused on the development and application of state-of-the-art multidisciplinary and multiphysics methods for predicting structural and mechanical properties of alloys based on *ab initio* thermodynamics. Aiming at multiscale metallurgical and engi-

neering problems, theoretical tools based on fundamental quantum-mechanics are combined with higher-scale (e.g. continuum linear-elasticity [25] or FEM [26]) approaches in a close cooperation with experimental groups and departments at the MPIE. The research topics cover mainly two areas (i) "materials-on-demand" design of new metallic alloys with tailored materials properties using quantum-mechanical methods and (ii) the development and application of multiscale methodologies in order to obtain a deeper understanding of existing phenomena and observed metallurgical trends.

The theory-guided materials design was successfully applied in the development [10] and processing simulations [27] of new biocompatible Ti-Nb implant materials for medical applications, compositional and structural optimization of Mg-Li alloys [28] for ultra light-weight applications (see also p. 139), or functional improvement of Fe<sub>3</sub>Al-based materials (see p. 131). The first two research topics have been performed in close cooperation with the MU department (Prof. Raabe), the third with the WT department (Prof. Frommeyer). The approach employed in these studies has proven to be highly versatile and has been utilized to address a number of materials science issues.

**The interaction of C and C-pairs in  $\alpha$  and  $\gamma$  iron** was one example, where this approach has been efficiently employed (O. Kim, Fig. 8). Based on these studies it was possible to accurately reproduce the large difference in C-solubilities in  $\alpha$  and  $\gamma$  iron and to explain this as a consequence of an intricate interplay between the short-ranged chemical and elastic parts of the C-interstitial formation energy as the primary reason [12].

In addition to problems where short range interactions dominate (which are in a DFT-approach straightforward to compute) also strain-induced

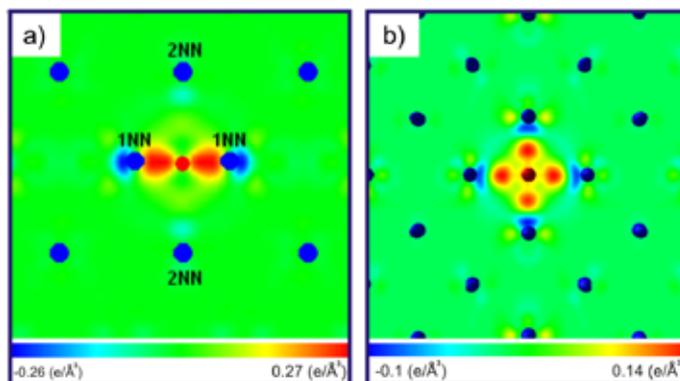


Fig. 8: Theoretically determined changes of the electronic density induced by C interstitials [12] in either  $\alpha$ -Fe (a) or  $\gamma$ -Fe (b) matrix (the sum of densities of a single C atom and that of pure Fe matrix is subtracted from the electronic density of the Fe-C system).



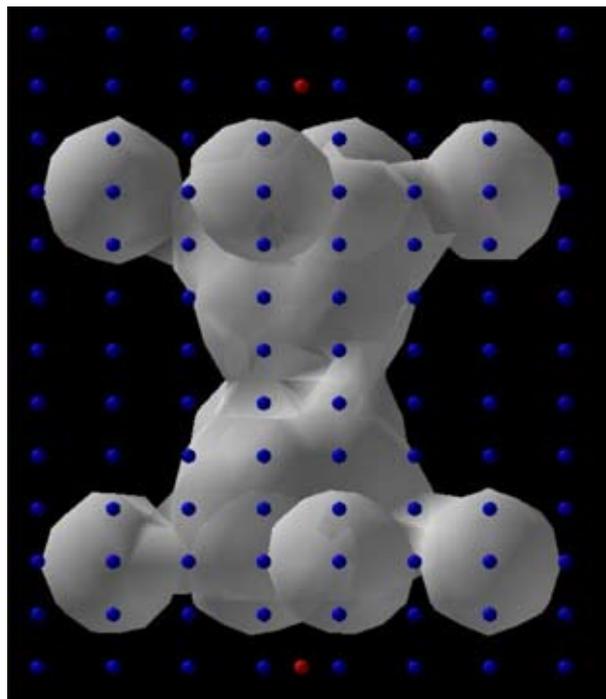
long-range interactions between interstitial impurity atoms in the Fe matrix have been studied (A. Udyansky, Fig. 9). This investigation has been performed in cooperation with Prof. Bugaev from MPI-MF in Stuttgart. The microscopic elasticity theory, i.e., the analytic Krivoglaz-Kanzaki force concept is employed. Here, the Kanzaki forces and the lattice Green's function are expressed through concentration expansion coefficients of the host lattice and the frequencies of lattice vibrations. The parameters of this phenomenological model are obtained from both *ab initio* calculations and atomistic modelling employing EAM potentials. The accurate description of the interatomic interactions achieved with this approach provides key quantities for the prediction of structural and thermodynamic properties of interstitial binary alloys.

**The study of biological materials** with an elaborated microstructure was another example when a multiscale approach proved to be very valuable. This study with the focus on  $\alpha$ -chitin was motivated by trying to analyse how the interplay of atomic structure and microstructure results in extremely strong and light-weight materials as e.g. the shells of lobsters. The ground-state atomic structure has been identified employing a hierarchical computational strategy in a combination with a new "quasi-static" approach to loading-conditions simulations (M. Petrov, together with the Microstructure group of L. Lymperakis).

The methodological hierarchy employed (i) less accurate but very fast computational schemes, as e.g. molecular dynamics, allowing an examination of millions of different atomic geometries, as well as (ii) more accurate DFT or DFT-based self-consistent tight-binding methods that have been applied to the most prospective atomic configurations pre-selected by the less accurate preceding simulations.

After determining the ground state structure, elastic properties of the single-crystalline  $\alpha$ -chitin were analysed. The results revealed a strong elastic anisotropy that could be linked to differences in the interatomic bonding character (hydrogen vs. covalent bonds). In a close cooperation with the MU department (Prof. Raabe) and the WS department (Prof. Pyzalla), the thus determined single-crystalline elastic constants were used in (i) experimental X-ray studies to determine the strain states in external and internal lobster's cuticle and (ii) a multi-scale homogenization study to describe the mechanical properties of a cuticle treated on the macroscale as a complex hierarchical composite by using *ab initio* determined atomic-scale input (see p. 169).

**The *ab initio* study of the bcc-hcp pressure-induced phase transition** in pure Fe performed in a cooperation with Prof. Šob from the Masaryk University and the Institute of Physics of Materials of the Academy of Sciences, Brno, Czech Republic,



**Fig. 9** Theoretically calculated isosurface of the interference part of the strain field in the  $\alpha$ -Fe matrix (ideal atomic positions are visualized by blue spheres) due to the presence of two C atoms (red spheres).

resulted in the identification of a complex interplay between different magnetic and structural phases along the bcc-hcp transformation path [29]. The project has been a continuation of on-going research activities focused on elastic properties of materials and magnetic and structural phase transformations [13,30,31].

### Microstructure (L. Lymperakis)

The focus of the microstructure group is on the atomic scale mechanisms which determine the evolution and the formation of the microstructure. The aim of the group is (i) to identify and quantify these mechanisms as well as to provide a deeper understanding and a consistent description of these mechanisms and (ii) to provide information not directly accessible by experiment. The various activities of the group are listed in the following:

**The mobilities of extended defects** such as Grain Boundaries (GBs) and dislocation determine the macroscopic relevant properties of functional materials to a large extent. The dynamical properties of dislocations and their dependence on the presence of foreign species such as hydrogen are strongly connected with the plastic behaviour of metals. Moreover, the mobility of GBs is the key mechanism which determines the microstructural evolution during growth. It controls the processes of recovery, recrystallization and grain growth and therefore

determines the grain size and subsequently the yield strength in the post-grown material.

In the first step the mobility of single edge type dislocations in Al has been considered. This step also served as a validation test for the EAM potential which was used in the subsequent GB mobility calculations. Molecular Dynamic (MD) calculations have been performed for large system sizes containing a single dislocation with its line lying along the [112] direction. The Peach-Koehler force was introduced to start and sustain the dislocation mobility. In line with previous studies three regimes in the mobility were observed: For the lowest driving forces the dislocation does not move at all, due to the Peirls barrier, while for larger driving forces an inverse relationship between the mobility of the dislocation and the applied temperature was found, reflecting the effect of thermal phonon interactions. For even larger stresses a plateau velocity was reached, which was found to be essentially independent of the applied stress and temperature. No transsonic velocities have been observed, as the regime of extremely large driving forces required for the observation of such dislocation velocities has not been considered.

The dynamics of GBs have been addressed in terms of molecular dynamics as well as in terms of the generalized stacking fault energy ( $\gamma$  surface). The aim of this project is not only to provide quantitative information about the GB mobility, but moreover to identify those atomic-scale mechanisms which control the GB mobility such as coordinated or uncorrelated atomic shuffle motion, interface kink formation, and/or lattice and secondary dislocation mobility. A major obstacle in the molecular dynamics description of the GB mobility is the introduction of a suitable driving force. While various approaches are available in the literature, not all of them are able to sustain the motion for any kind of GB and/or to describe the dependence of the GB mobility on any of the five relevant misorientation and boundary inclination degrees of freedom. Therefore in the first stage of the project the different approaches are tested and the validity and suitability of the

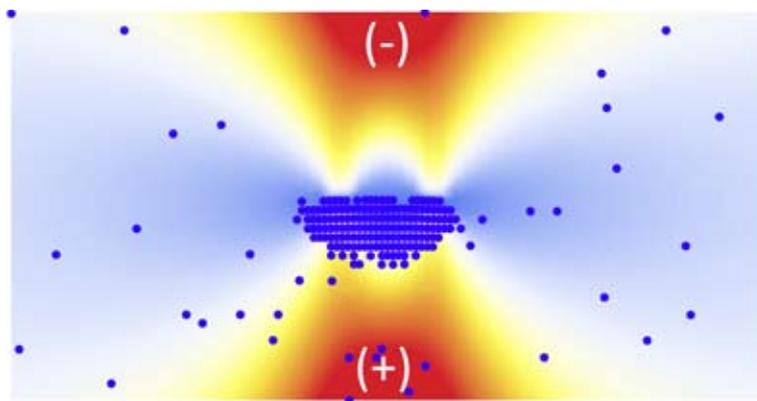
corresponding driving forces are considered. The  $\gamma$  surface constitutes an alternative approach to investigate the GB dynamics. The advantage of this approach is that it directly combines the atomistic scale calculations with continuum mechanics. In a joined project with the ICAMS 'Micromechanical and Macroscopic Modelling' department, the gamma surfaces of various special Al GBs have been calculated using EAM potentials.

The mechanical properties of functional materials depend strongly on the presence of foreign chemical species: Tiny quantities of impurities can drastically alter the mechanical response of metals. It is well established by now that hydrogen is responsible for structural degradation in functional materials, a phenomenon which particularly affects high-strength steels. The H-enhanced local plasticity (HELP) mechanism has attracted considerable interest recently.

**The hydride formation and the influence of interstitial H** on the dislocation strain field in a Ni matrix have been investigated (see p. 209). A hierarchical approach which combines first principles, EAM potentials, and Monte Carlo calculations based on a lattice gas Hamiltonian has been developed in order to efficiently and accurately describe the corresponding length scales: Short-ranged H-H and H-metal interaction, large-scale elastic interactions, and the huge configurational space characterize the H incorporation in the dislocations strain field.

The calculations revealed the strong tendency of H to cluster and form hydride phases in Ni and to preferentially incorporate into the tensile regions of the dislocations strain field (see Fig. 10). The observed formation of local hydrides in the tensile strain field around dislocations contradicts the original HELP mechanism. However, the calculations also revealed that the H incorporation introduces a reduction in the dislocation-dislocation interaction, in line with the HELP mechanism. The calculated dependence of the H incorporation on the H chemical potential indicated a hydrogen pressure threshold for

a dramatic increase of the concentration of the incorporated hydrogen. This finding raises the interesting question whether the observed homogeneous hydride nucleation might be exploitable for hydrogen storage applications. In particular, hydrogen could be introduced into the metal by exposing the metal to a sufficiently large  $H_2$  pressure or by means of electrochemical charging and would subsequently be released due to the destabilisation of the hydride nuclei as the H chemical potential decreases in response to a decrease in the ambient H pressure, which has the obvious advantage of negligible desorption energies.



**Fig. 10:** H atoms (blue balls) in the stress field of a partial dislocations dipole. The contour plot indicates the  $\sigma_{xx}$  component of the total stress field. The H preferentially incorporates in the tensile regions of the dislocations strain field.



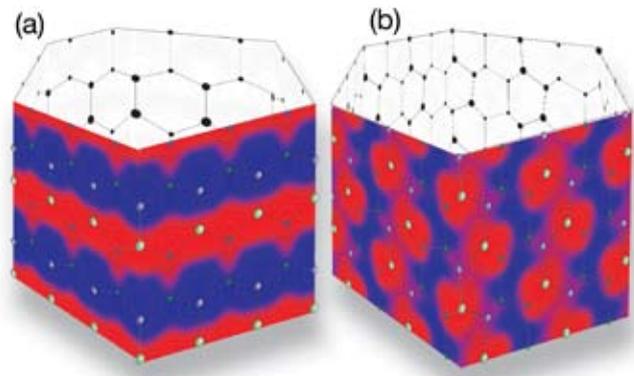
### Group-III-nitride based semiconductors

are widely used materials in the optoelectronics industry for short wavelength photonic applications as well as for high frequency high power devices. The group has an extensive experience on these materials and had under its supervision two projects funded by EU and the DFG.

The growth of semiconductor nanowires is a rapidly expanding field since they constitute the promising building blocks for nanoscale electronics. One of the inherent characteristics of the nanowires is the rather large surface/volume ratio. Hence, surface phenomena are expected to control to a large extent the properties of the nanowire such as the shape and the axial/radial ratio. Therefore, in order to achieve improved device features a detailed understanding of the growth mechanisms is crucial.

The majority of reported GaN nanowires (NW) grow along the polar [0001]-direction exposing as side facets the *m*- and *a*-plane non-polar surfaces. Thus, the thermodynamics and the adatom kinetics on the non-polar GaN surfaces have been studied by *ab-initio* calculations. Our calculations revealed that both non-polar surfaces stabilize the stoichiometric Ga-N dimer configuration for growth under N-rich conditions which are relevant for the growth of NWs. In the next step the Ga adatom kinetics on both non-polar surfaces has been considered. For symmetry reasons and the stoichiometric nature (presence of both anions and cations) of these surfaces, the kinetic barriers are anisotropic: On the *m*-plane surface Ga adatoms exhibit larger diffusion lengths in the lateral NW directions, while the situation is reversed on the *a*-plane surface where the diffusion barriers are considerably smaller for diffusion along the axial direction (Fig. 11). A consequence of these results is that the diffusion induced mechanism (in which the NW growth proceeds via adatoms diffusion along the side facets to the top surface) is more pronounced for the *a*-plane with respect to the *m*-plane surfaces. On the other hand the calculations revealed a striking result for the N adatoms: For a number of different lateral positions located close to the N surface atoms, the N adatom attracts the N surface atom, and desorbs as a nitrogen molecule instead of adsorbing on the surface. Thus, the non-polar GaN surfaces are intrinsically unstable against atomic N [32].

Although the calculations have been applied for a semiconductor, the underlying formalism and methodology is universal and ready to be applied in metallic systems: The thermodynamics and the kinetics of metallic void surfaces (e.g. relevant in diffusion bonding techniques) is a characteristic example.



**Fig. 11:** Perspective and schematic view of the potential energy surface for Ga adatom diffusion on the side facets of GaN NWs. The side facets consist of (a) *m*-plane and (b) *a*-plane surfaces.

**Semiconductor nanostructures** such as nanowires, quantum wells, and quantum dots, constitute key features in the design of novel high-performance optoelectronic devices. A crucial aspect in the design and optimization of such devices is a detailed and predictive understanding of the optical and electronic properties of the relevant nanostructures. As discussed above, the surface thermodynamics and kinetics of nanostructures can be directly addressed by *ab initio* calculations. However, since typical nanostructures consist of  $10^4 \dots 10^7$  atoms, a direct calculation of their electronic and optical properties is not feasible employing DFT. Therefore, based on the S/PHI/nX library, an alternative approach – a plane-wave implementation of the k.p-formalism – has been developed. This approach allows an efficient calculation of the electron and hole bound and wetting layer states and the corresponding Coulomb matrix elements (O. Marquardt, T. Hickel) [15]. Moreover, nanostructures such as quantum dots are often highly strained. Thus, strain and piezoelectric effects had to be explicitly taken into account. Therefore, the package has been extended such that elastic distortions can be determined by minimizing the strain free energy [33]. These methods have been successfully applied to various semiconductor nanostructures of zincblende and wurzite crystal structure.

**Dilute nitride semiconductors** such as GaAs or InAs with a few percent nitrogen are highly promising materials for optoelectronic and photovoltaic applications. A major challenge in fabricating such alloys is the extreme low equilibrium bulk solubility of N. Therefore, it has been studied whether and under what conditions surface kinetics may be employed to enhance the nitrogen concentration and to kinetically stabilize alloy compositions far away from thermodynamic equilibrium. Using *ab initio* computed

surface phase and surface solubility diagrams it has been shown that the nitrogen concentration can be enhanced by several orders of magnitude [34]. Based on similar calculations it has been also demonstrated that in contrast to common believe solubility cannot be simply enhanced by adding a further species (such as In in GaAs) which induces strain of the opposite sign and has been thus expected to relieve the strain induced by the nitrogen [14] (H. Abu-Farsakh).

### Defect Chemistry and Spectroscopy (C. Freysoldt)

The group “Defect Chemistry and Spectroscopy” was established in September 2007. Its task is to study atomic-scale defects in non-metallic materials such as oxides, electrolytes, or semiconductors by means of *ab initio* methods, and to develop state-of-the-art methods and concepts for this purpose.

The importance of intrinsic point defects (e.g. vacancies, interstitials, or anti-site atoms) and impurities arises from their ability to modify or even determine the electrical, chemical, and optical properties of these materials. Even low concentrations in the order of  $10^{-2}$  to  $10^{-6}$  may exert a significant influence. Therefore, they are practically ubiquitous and must be taken into account to understand the behaviour of real materials. Yet, the low concentrations are a challenge to quantify the relevant microscopic parameters, such as atomic structure, formation energy, electrical and chemical activity, mobility, etc. Among the available experimental techniques, spectroscopic methods are particularly useful to detect and analyse the defects, albeit the indirect information obtained often needs a careful interpretation. Here, *ab initio* theory can step in and guide the interpretation of experimental findings, as well as provide quantities that are unmeasurable or hard to obtain from experiment.

Many defect-specific methods, both experimental and theoretical, have emerged in the field of semiconductors for (opto)electronic devices. Modern technology allows to control the structure and composition of samples to a great degree, which is extremely helpful to advance the agreement between theory and experiment. Transferring the established methods to materials relevant in steel technology, notably oxides, protective coatings, or electrolytes, promises a significant boost in understanding their electrical, chemical, and transport characteristics. The group’s aim is to become active in both directions: developing state-of-the-art methods and applying them to problems in, e.g., adhesion and corrosion.

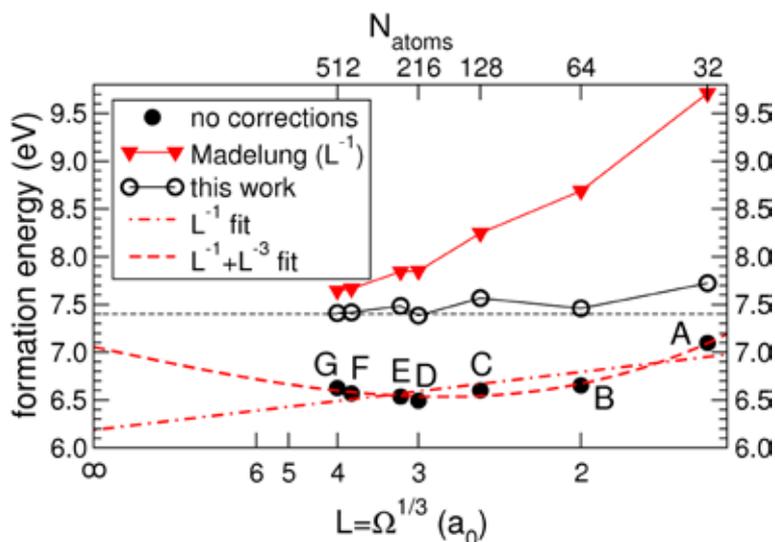


Fig. 12: Formation energy of +2 vacancy in diamond in the supercell approximation: The *ab initio* correction scheme (empty spheres) shows an improved convergence compared to empirical schemes.

The major computational challenges in describing oxides, electrolytes etc. are rooted in the supercell approximation and the band gap problem of DFT. On the one hand, the single defect must be approximated by a periodic array of defects in order to employ the highly optimized and well tested computer codes for solids. This supercell approximation induces artifacts, notably for charged defects, requiring large supercells to reduce their magnitude, and often additional corrections for electrostatic interactions. In cooperation with the Algorithm Design and Modelling group (S. Boeck), the S/PHI/nX package is optimized to meet these demands. In this context, improved minimization schemes and projection techniques for large-scale calculations were developed and implemented. With these, a significant reduction of the memory consumption and CPU times needed for large calculations was achieved, shifting the size limit from ~200 atoms to roughly 1000 atoms on a single node. Moreover, the existing correction schemes for charged defects were analysed in the framework of microscopic linear theory of electrostatics. From this analysis, a novel, fully *ab initio* correction scheme was developed that gets along without empirical parameters or fitting procedures [35].

On the other hand, standard DFT fails to accurately describe band gaps, with typical errors of 30-100%. For the electrical activity of a defect, however, the energetic position of the defect levels with respect to the band edges of the surrounding matrix is decisive. Their prediction in DFT is a difficult task and demands a thorough understanding of the defect character (e.g. deep or shallow defects) and the relevant physics. Improved functionals like EXX and theories beyond DFT, such as the GW approach, are highly attractive developments to resolve these difficulties for defects. However, the computational effort for these methods



is still prohibitive for supercells with dozens or even hundreds of atoms. Sufficiently efficient algorithms emerge only slowly. The group is planning to play an active role in this area (B. Lange).

A further activity is the calculation of spectroscopical parameters that allow to link experimental observations with valid microscopic structural models. In the EPR-Solar project, electron paramagnetic resonance (EPR) spectroscopy is used to identify and analyse degradation mechanisms in Si-based solar cells. The defect group is implementing algorithms to compute the spectroscopic hyperfine and Zeeman parameters, which will then be used to analyse structural models of the “Si dangling bond” defect (G. Pfanner).

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- Young, T.D.; Marquardt, O.: phys. stat. sol. b (2008) *accepted*.
- Abu-Farsakh, H.; Neugebauer, J.: Phys. Rev. B (2008) *submitted*.
- Freysoldt, C.; Neugebauer, J.; Van de Walle, C.: Phys. Rev. Lett. (2008) *submitted*.

## Research Projects in Progress

### Algorithm Design and Modelling

*Boeck, Neugebauer:* Development of an automatic MPI parallelization technique for electronic structure calculations

*Bubnik, Boeck, Neugebauer:* Development of methods for interactive scalar and multi-dimensional vector field visualization

*Uchdorf, Boeck, Neugebauer:* Development of an ontology-based database model for multi-physics applications

### Computational Phase Studies

*Aydin, Ismer, Hickel, Neugebauer:* Chemical trends for the solubility and diffusion of hydrogen in metals, *in collaboration with Drautz\** (\*ICAMS Bochum)

*Dick, Hickel, Neugebauer, Raabe:* Ab initio calculation of free energies, stacking-fault and grain-boundary energies at finite temperatures in Fe-Mn-C alloys, *in collaboration with Dronskowski\*\*, Hallstedt\*\* et al. (RWTH Aachen)*

*Grabowski, Hickel, Neugebauer:* Ab initio based modelling of thermodynamic properties of metals close to the melting point, *in collaboration with Neuhaus\** (\*FRM-II Garching)

*Grabowski, Hickel, Neugebauer:* Ab initio determination of Helmholtz free energies and derived properties (phase diagrams, heat capacities, thermal expansions) for Al alloys containing Si, Mg and Cu, *in collaboration with Hallstedt\*, Egrý\*\*, Schmid-Fetzer\*\*\*, Rettenmeyer\*\*\*\* (RWTH Aachen, \*\*DLR Köln, \*\*\*TU Clausthal, \*\*\*\*U Jena)*

*Grabowski, Hickel, Neugebauer:* Kinetic, structural and dynamic properties of the phase change alloy GeSb<sub>2</sub>Te<sub>4</sub>, *in collaboration with Lencer\*, Wuttig\** (\*RWTH Aachen)

*Hickel, Neugebauer:* Development of tight-binding approaches and bond-order potentials for an atomistic description of magnetic materials, *in collaboration with Drautz\** (\*ICAMS Bochum), *Elsässer\*\** (\*\*IWM Freiburg)

*Hickel, Neugebauer:* Prediction of structures and phase diagrams in solid state chemistry, *in collaboration with Jansen\*, Schön\** (\*MPI-FKF, Stuttgart)

*Ismer, Hickel, Neugebauer:* Influence of defects on the energetics and dynamics of hydrogen in manganese steels

*Körmann, Dick, Hickel, Neugebauer:* Ab initio simulation of magnetic contributions to the thermodynamics

of metals, *in collaboration with Hallstedt\** (\*RWTH Aachen)

*Marquardt, Hickel, Neugebauer:* Multiscale growth and doping simulations of nanostructured devices, *in collaboration with Jahnke\*, Czycholl\** (\*Univ. Bremen)

*Ujttewaal, Hickel, Neugebauer:* Ab initio investigation of temperature dependent effects in shape memory Heusler alloys, *in collaboration with Ziebeck\*, Neumann\** (\*Loughborough University, UK), *Entel\*\** (\*\*Univ. Duisburg-Essen), *Petry\*\*\*, Neuhaus\*\*\** (\*\*FRM-II Garching)

### Ab initio Thermodynamics

*Friák:* Martensitic phase transitions in materials, *in collaboration with Legut\** (\*Uppsala University)

*Friák:* Ab initio study of transition-metal disilicides, *in collaboration with Šob\** (\*Masaryk University, Brno)

*Friák, Counts, Raabe, Neugebauer:* Compositional and structural optimization of Mg-Li alloys for ultra light-weight applications

*Friák, Deges, Krein, Palm, Stein, Frommeyer, Neugebauer:* Scale-bridging multi-disciplinary approach to the integral elastic response of Fe<sub>3</sub>Al-based alloys

*Friák, Hassel, Neugebauer:* Ab initio study of the Re nanowires

*Friák, Ma, Raabe, Neugebauer:* Ab initio study of the solid solution hardening in Al

*Friák, Neugebauer:* Scale-bridging studies of the elastic contributions to nucleation and initial microstructure formation in the eutectic Ti-Fe system, *in collaboration with Emmerich\*, Eckert\*\** (\*RWTH Aachen, \*\*IFW Dresden)

*Friák, Neugebauer:* Ab initio study of the pressure stabilized NiTi allotropes, *in collaboration with Holec\** (\*University of Cambridge), *Dlouhý\*\** (\*\*Institute of Physics of Materials, Brno)

*Friák, Neugebauer:* Structural and elastic properties of biomimetic calcite composites *in collaboration with Aichmayer\* and Cölfen\** (\*MPI of Colloids and Interfaces, Potsdam-Golm)

*Friák, Sander, Ma, Counts, Raabe, Neugebauer:* Ab-initio based study of the elastic properties of dual-phase Ti-Nb polycrystalline composites

*Udyansky, Friák, Neugebauer:* Ab initio based statistical-thermodynamics analysis of the Fe-Co system, *in cooperation with Diaz-Ortiz\* and Bugaev\** (MPI-MF, Stuttgart)



## Microstructure

*Lymperakis, Neugebauer:* Band alignment of ScN and GaN semiconductors, *in collaboration with Holec\** (\*University of Cambridge)

*Lymperakis, Neugebauer:* Ab-initio based growth of novel III-Nitride nanostructures

*Lymperakis, Neugebauer:* High efficient green and yellow light emitting diodes

*Lymperakis, Neugebauer:* Deep Ultraviolet Light Emitting Diodes on AlN Substrates: Doping Strategies and Epitaxial Growth

*Petrov, Lymperakis, Neugebauer:* Third order elastic constants of III-Nitride alloys

*Petrov, Lymperakis, Neugebauer:* Theoretical Study of Strain Effects on ELNES and Electronic Structure of AlGa<sub>N</sub>, *in collaboration with Holec\** (\*University of Cambridge)

*von Pezold, Lymperakis, Neugebauer, Raabe:* Multi-scale approach to describe the effect of H on dislocation mobility and interaction

*von Pezold, Lymperakis, Neugebauer, Raabe:* Mobility of grain boundaries

## Defect Chemistry and Spectroscopy

*Freysoldt, Neugebauer:* Electronic structure at interfaces using DFT and quasiparticle corrections

*Freysoldt, Neugebauer:* Simulation of intermolecular forces in polyelectrolyte with embedded Montmorillonite nanoclay, *in collaboration with Grundmeier\** (\*University of Paderborn)

*Lange, Freysoldt, Neugebauer:* Development and implementation of highly efficient EXX calculations for defects

*Lange, Freysoldt, Neugebauer:* Generation of optimal LCAO basis sets

*Lange, Freysoldt, Neugebauer:* Co-doping and passivation of hydrogen in GaN-based light-emitting devices

*Pfanner, Freysoldt, Neugebauer:* Defect structures and EPR parameters in Si-based solar cells

## Joint group activities

*Abu-Farsakh, Neugebauer:* Growth simulations of thermodynamically highly unstable nitride based alloys, *in collaboration with Albrecht\** (\*IKZ Berlin)

*Biedermann, Petrov, Friák, Lymperakis, Raabe, Neugebauer:* Theoretical study of the  $\alpha$ -chitin NMR spectra

*Counts, Grabowski, Friák, Hickel, Neugebauer:* Phonon spectra of Li and Li-based alloys

*Friák, Hickel, Neugebauer:* Quanten-mechanische Bestimmung der Phasenstabilität und Diffusion in hochtemperaturbeständigen ferritischen Stählen

*Friák, Körmann, Grabowski, Udyansky, Hickel, Neugebauer:* Ab initio study of the  $\alpha$ -iron phase stability limits, *in collaboration with Černý\** (\*Brno University of Technology)

*Petrov, Friák, Lymperakis, Neugebauer, Fabritius, Nikolov, Raabe:* Hierarchical modelling of the elastic properties of lobster cuticle from nano- to macroscale

*Petrov, Friák, Lymperakis, Neugebauer, Pinto, Pyzalla, Raabe:* Internal stresses in the exoskeleton of a homarus americanus Lobster: A combined experimental and theoretical study

*Todorova, Hickel, Neugebauer:* Ab initio study of corrosion: Adsorbate phases on surfaces and phase diagrams

*Lymperakis, Marquardt, Freysoldt:* Electric activity of charged edge type dislocations in GaN

*Udyansky, Friák, von Pezold, Lymperakis, Neugebauer:* Strain-induced long-range inter-actions in interstitial solid solutions, *in cooperation with Bugaev\** (\*MPI-MF, Stuttgart)





# Department of Interface Chemistry and Surface Engineering

M. Stratmann

## Introduction

The department mainly focuses on chemical reactions and physical properties of surfaces and interfaces with particular emphasis on finding ways how to increase their stability and to include functional properties into coatings on structural materials. The material classes of particular interest include metals, polymers, ceramics and their composites. Scientific studies related to these materials in particular concentrate on degradation reactions such as aqueous corrosion, high-temperature corrosion, tribocorrosion and de-adhesion reactions with the aim to understand their underlying physico-chemical reaction mechanisms. Based on this knowledge new and superior surface and interfaces are designed, characterised by their novel chemical composition, morphology and molecular and atomistic structure. To a great extent this requires the application of new surface modification techniques. The physico-chemical understanding of such technologies with regard to their application in tailoring surfaces and interfaces complements the analytical approach of the department.

Currently, the department includes approximately 70 people and among them more than 50 scientists working on more than 60 projects – most of them integrated in international collaborations with scientists in Europe, Asia and the United States. The experimental equipment is exceptional even on an international scale and allows performing state of the art science in surface and interface chemistry.

The structure of the department is summarised in Fig. 1. This structure includes three major lines:

- i. *Scientific groups* as the most important part of the management structure. These groups are competence centres in various areas of science and they include specific experimental and theoretical knowledge. The scientific groups are largely integrated in the major departmental fields of scientific interests; however in parallel they also have their own specific and unique scientific goals.

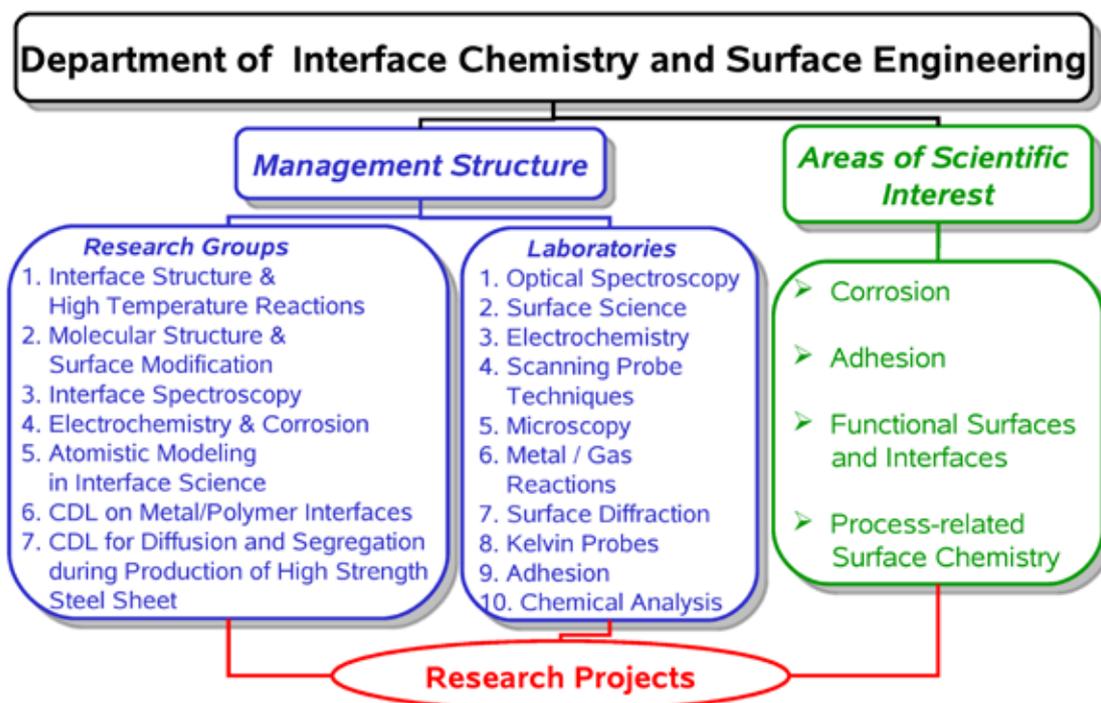


Fig. 1: Organization of the Department of Interface Chemistry and Surface Engineering.

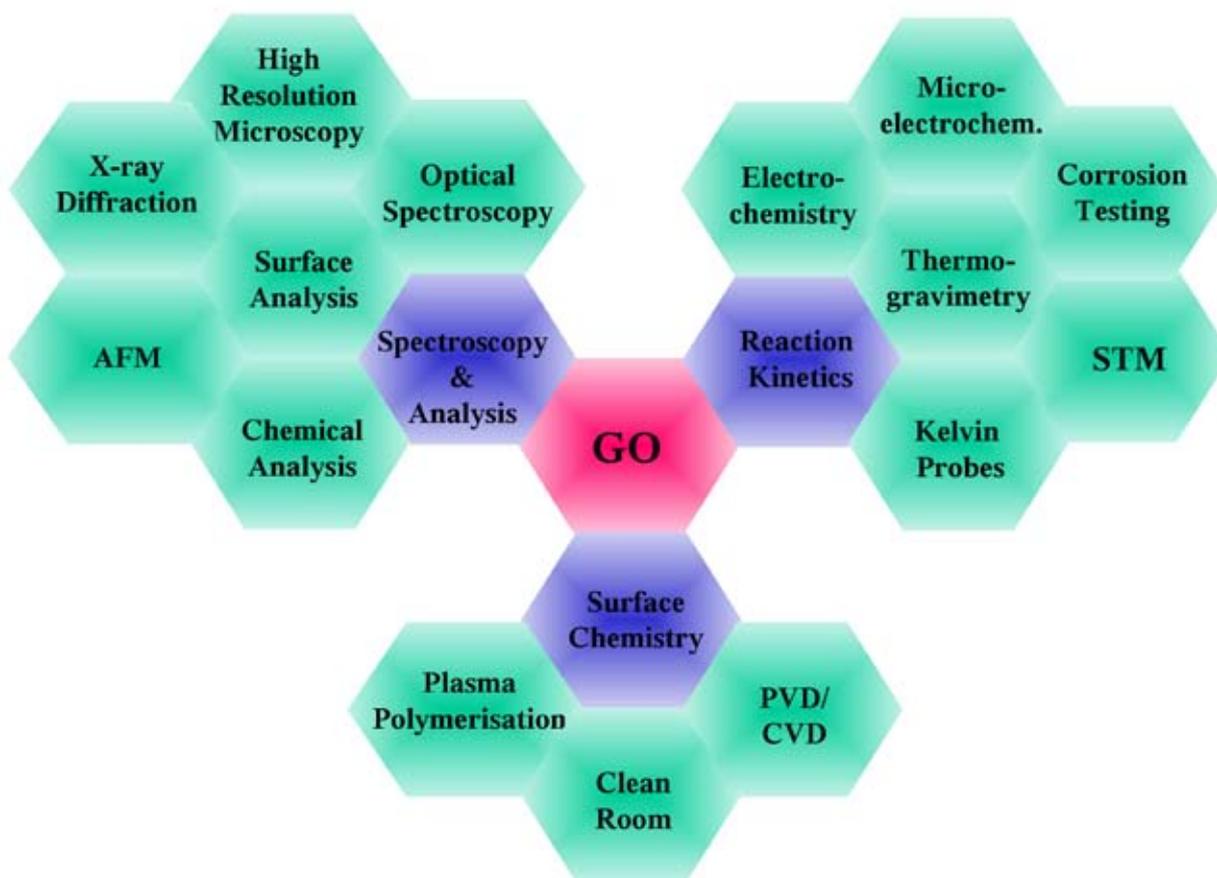


Fig. 2: Experimental laboratories of the Department of Interface Chemistry and Surface Engineering.

- ii. Laboratories (Fig. 2) include the experimental techniques necessary for investigating surfaces and interfaces. Besides serving the scientific groups for their experimental needs they serve as centres for the development of new and often unique experimental set-ups as a prerequisite for many state of the art research projects. Obviously, there exist strong links between the specific experimental knowledge of the scientific groups and some of the key laboratories.
- iii. The department has clearly defined *areas of scientific interests* which are common for all scientific groups in the department and which define a scientific link between them. Indeed, the long term goals on which the areas of scientific interests are based, can be achieved only if the groups of differing scientific knowledge strongly collaborate. The collaboration is organised in short term projects typically financed either by scholarships or by European or German funds.

The department is well integrated into the scientific structure of the institute. Major links include aspects

of micromechanics of surfaces, interfaces and thin films (Prof. Raabe), the correlation between the reactivity of surfaces and their microstructure (Prof. Raabe), the corrosion stability and surface characteristics of novel materials after heat treatment and the formation of novel microelectrode arrays (Prof. Frommeyer), and an intense collaboration with Prof. Neugebauer in areas such as hydrogen in steel or semiconducting oxides. The atomistic scale of many experiments within the department is very much in line with the computational expertise of Prof. Neugebauer's department.

Finally, it should be mentioned that the department is highly dedicated to activities beyond the institute. The department coordinates a number of European research projects, and has attracted two *Christian Doppler (CD) Laboratories* financed by the Austrian CD Society: the *CD lab on Polymer/Metal Interfaces* (Head: Prof. Dr.-Ing. Guido Grundmeier) and the *CD lab on Diffusion and Segregation Mechanisms during Production of High Strength Steel Sheet* (Head: Dr. Michael Rohwerder). Furthermore, the department shows a strong presence in the IMPRS "SurMat".



## Scientific Concepts

All scientific groups are horizontally linked by common areas of scientific interest:

### Corrosion

Research of the department includes aspects such as aqueous corrosion, atmospheric corrosion, flow induced corrosion, microbially influenced corrosion, corrosion inhibition and high temperature corrosion. Aspects which have been of considerable importance for the department over the reporting period 2007 and 2008 years are:

- *Corrosion studies of novel materials with considerable technological importance:* in the reporting period the focus was mainly on shape memory alloys [1-3]. Another focus is the development of novel experimental tools for corrosion investigation, such as a new device for performing accelerated cyclic immersion tests [4], which also helps in the development of novel materials.
- *Understanding of corrosion reactions on a micro-structure scale:* the activities focussing on the influence of the grain orientation and the grain boundary structure on local corrosion kinetics have been intensified [5]. Besides scanning droplet cell, SECM (Scanning Electrochemical Microscopy) will play here a crucial role. In cooperation with Prof. Schuhmann from the RUB it is planned to combine SECM and SKPFM (Scanning Kelvin Probe Force Microscopy). Although contrary to what is found in the majority of publications about the application SKPFM in corrosion science, it generally is not providing information about localised electrochemical activity [6], the results obtained by Kelvin probe techniques still provide valuable information about the electrochemical behaviour under the conditions of measurement [7]. The development of special microelectrodes is another activity of importance [8].
- *Understanding of corrosion reactions from an atomistic to a macroscopic scale:* the focus in the reporting period was mainly on alloy corrosion, where the new group of Dr. F. Renner was successfully integrated right from its start, bringing valuable expertise in synchrotron based x-ray diffraction [9] into the department. Another focus point was degradation of organic monolayer films.
- *Optimisation of corrosion resistant surfaces by combinatorial materials science.* Graded alloy surfaces are being prepared using multi-component PVD techniques and subsequently

studied by localised fast screening techniques. For aqueous corrosion these are scanning electrochemical droplet cell and for atmospheric corrosion the scanning Kelvin probe [10].

- *Combination of experiment and theory* will be of steadily increasing importance as a powerful tool for designing novel materials concepts. However, properties such as corrosion performance are based on extremely complex processes. Such complex reaction and the correlated complex environments are still a problem for theoretical approaches. So far, complementing the experimental studies, two fundamental aspects of paint delamination have been investigated by theoretical methods:
  1. The electrochemical oxygen reduction reaction (ORR), as cathodic partial reaction, was studied to elucidate the step-by-step mechanism, its intermediates, their stability and reactivity towards organic molecules. Reactive intermediates as, e.g., hydroxyl radicals, may lead to a chemical degradation of the coating.
  2. The galvanic coupling in delamination is due to a facile migration of ions along or near the interface. In order to gain insight into this phenomenon, ion diffusion was studied in a model interface with well defined structure.

### Adhesion

As a consequence of the invention of the Scanning Kelvin Probe (SKP) by M. Stratmann and co-workers, the study of buried metal/oxide – polymer interfaces is a prime target of the department. This technique has proven to be most powerful in studying even elementary reactions which lead to de-adhesion of polymers from solid surfaces in aggressive media.

Research concentrates on the following issues:

- *New analytical instruments.* New combined experimental and computational approaches to understand and exploit the surface enhancement mechanisms near small metal structures are currently being developed. These may help to conduct spectroscopic experiments in-situ during single-molecule adhesion studies. For these, a detection of conformational changes will substantially enrich the understanding of the atomic level of de-adhesion.
- *Mobility of water and ions along the buried interface.* Here the main focus was on model interfaces between well defined molecular monolayers on inert substrates and model polymers. So far the investigations have mainly been performed by SKP. While this is a powerful

technique for in-situ monitoring the movement of ions along the interface, it does not directly provide information about the concentration of ions at the interface. Hence, a combination with optical spectroscopy is currently implemented in the new group of Dr. A. Erbe.

- *Electrochemical and chemical reactions at the buried interface leading to de-adhesion.* Based on our findings that inherently stable interfaces can be designed by optimising the oxide defect structure such that the work function of the oxide covered metal/polymer interface matches the work function of the freely corroding iron surface [7], also plasma based techniques have been applied to achieve adjusted potential adjustments. For instance, by using ultra-thin Si-organic plasma polymer layers the potential of zinc surfaces was adjusted in a way, that it became equal to the free corrosion potential of zinc in aqueous media [11].
- *Chemical bonds at the buried interface.* Obviously chemical bonds are essential for creating stable composites. The department had considerable success in the development of the self assembled monolayer concept for adhesion promoters used on technical surfaces. In particular phosphonate based molecules have been developed for Al-oxide terminated surfaces and a successful industrial surface treatment is based on the application of this technology (Chemetall). The modification of oxide covered aluminium surfaces by means of H<sub>2</sub>O-plasma treatments led to an increased hydroxyl density on these surfaces and thereby to a significantly increased adsorption kinetic of organ-ophosphonic acids [12].
- *Theoretical studies of the adhesion mechanisms.* Density functional methods are applied to elucidate the binding mechanism of paradigmatic examples for adhesion promoters, e.g., silanols and phosphonic acids on aluminium or zinc oxides/hydroxides and thiols on gold.

### Functional surfaces and interfaces

The department has a considerable interest in the study of new functional surfaces and interfaces aiming to combine traditional aspects of organic coatings such as adhesion or corrosion protection with novel aspects. Within the last two years physical and chemical vapour deposition as well as colloid chemistry have been of particular interest:

- *Plasma polymer composite films.* By combining PVD of Ag and plasma polymerisation of fluorinated monomers, model metal/polymer nanocomposite films could be designed which allow a detailed analysis of the release properties

of Ag-nanoclusters in a perfluorinated matrix. The process of electrochemical Ostwald ripening of silver nanoparticle monolayers within perfluorinated matrices could be observed. Processes on the surface of these nanoparticles within nanocomposite systems have a significant impact on the silver release properties of antibacterial Ag/polymer-nanocomposites [13].

- *PVD coatings.* These coatings are of considerable interest on steel and galvanised steel substrates. They are either used to form tailored oxides with a specific electronic structure or they are used for surface alloying. Currently a new multi-source evaporation chamber is planned for research on the development of highly corrosion resistant materials, based on a combinatorial approach.
- *Smart interfaces with self repair properties.* The focus here is on conducting polymers and inorganic nanocapsules with intelligent release function, either to be embedded in the polymer matrix or even within galvanic alloy deposited zinc coatings. For conducting polymers the concept of intelligent release has already successfully been shown to work and it is also quite well understood how well performing composite coatings should look like [14]. However, still problems with anodic metal corrosion driven by the conducting polymer itself have to be solved. As a first step SKP could be shown to be applicable as a tool for in-situ monitoring the corrosion reaction at the buried interface. For the case of inorganic nanocapsules, the incorporation of molecules into mesoporous silica particles and their encapsulation was successfully performed, as well as a fundamental study of their release from the mesoporous structure [15, 16]. However, the co-deposition of the particle during electro-galvanising proved to be difficult. Currently, fundamental research on the mechanism of co-deposition is carried out, as so far the knowledge reported in literature is mainly of empirical nature.
- *Switchable organic surface coating:* Materials which change properties with an externally triggered change in conformation or configuration can be used for making surfaces adaptive. Intelligent molecular design and surface chemistry need to be combined in order to use the observed effects in applications, which are attempted in future collaboration with organic chemists.

### Process-related surface chemistry

Steel surfaces are of particular interest for the department. In close collaboration with the steel industry and in many cases funded by European



funds steel related projects cover the full range of surface treatments which are important in nowadays steel surface chemistry.

- Short time annealing is studied to understand the scale formation prior to galvanising in oxidizing and reducing atmospheres. The segregation of non-metallic elements is of particular importance [17,18].
- Hot dip galvanizing is determined by wetting of liquid zinc on the oxide covered surface and by interfacial reaction between the liquid bath and the substrate. Both phenomena are studied with unique instrumentation [19].
- Cleaning of steel and the role of remaining residual salt is a topic of significant importance for steel industry [20-22]
- Adhesive bonding as one of the most promising joining technologies for multimaterials applications is studied with the focus on the understanding of the stability of interphases under corrosive and mechanical load.

## Scientific Groups

### Molecular Structure and Surface Modification (M. Rohwerder)

The main scope of this group is to address fundamental questions of surface and coating technology by isolating the crucial problems behind them and designing model experiments and model samples for their systematic investigation.

The main vision for the medium-term future is to provide a detailed model for electrochemically driven coating delamination (in cooperation especially with the simulation group of Dr. Blumenau/ Dr. Biedermann) and based on this to develop novel protection concepts. This requires splitting the problem into several sub-problems which are to be studied in detail, such as the role of the surface oxide layers and the molecular structure on electron transfer (ETR) during oxygen reduction at the buried polymer/metal interface, how to understand mobility of charged species along such interfaces or how degradation at these interfaces occurs in detail.

One main task of the group is to further develop and improve techniques crucial for addressing these questions. This includes the UHV cluster of the department which is continuously being improved, featuring a number of different surface preparation and analysis techniques, as well as the further development of the Kelvin Probe technique and its application to new fields.

According to the above the current main interests are focussed on:

- Elementary steps of electrochemically driven de-adhesion of organic coatings
- Semiconducting properties of surface oxide films
- Wetting and interfacial reactions at metal/metal melt interfaces
- Intelligent self-healing concepts for corrosion protection
- Surface modification by self-organised monolayers

- SKP as a tool for general application for processes at buried interfaces

In detail the following main activities on these research interests can be summarized

#### *i. Elementary steps of electrochemically driven de-adhesion of organic coatings*

A systematic investigation of electron transfer reactions at polymer/metal interfaces requires detailed knowledge of the molecular structure at the interface – which in most cases is not really understood on the atomistic scale. For this reason, the system alkane thiol/Au(111) was chosen as a well defined organic/metal interface.

Although thiol SAMs on gold is one of the best investigated systems, there are still many questions unanswered up to now. For instance, even for a perfectly self-organised monolayer there is still no generally accepted agreement on the exact position of the sulphur atoms on the gold surface. Even less information is available about defect structures, such as domain boundaries. However, our research showed that inside the potential window of interest for our studies the oxygen reduction reaction (ORR) is completely inhibited on the highly-organized monolayer within the well-ordered domains. All activity occurs solely in the domain boundaries and not in the well-ordered monolayer regions. Also, of course, the monolayer degradation is localised in these defective areas. While the localised degradation in these sites occurs relatively fast, the break-down of the highly resistive monolayer within the domains occurs only after extended exposure, but then very suddenly. This is most likely due to a radical driven chain reaction, which is also suggested by simulations performed in the group of Dr. Blumenau/Dr. Biedermann. Another interesting result is that even after desorption of the thiol monolayer still a very pronounced inhibition is observed. Our results indicate that the monolayer retains at least some of its properties and stays near the surface even in the desorbed state, and that this

desorbed monolayer still provides a significant barrier against oxygen permeation to the surface. This is of considerable interest, as here only the effect of oxygen transport is involved, ETR should not be hindered [23].

Since it is difficult to describe not well ordered structures, the fact that all reactivity is observed in the not well-organized regions significantly aggravates achieving the intended structure-reactivity correlation. Hence, in order to place our research on a broader basis, other thiol molecules are used (in cooperation with Prof. Wöll, RUB) which show lower inhibition of the ORR and, as first results suggest, not full inhibition on the highly ordered domains.

Another aspect which is of importance is the role of ion concentration at the interface. Preliminary experiments on critical salt levels show that threshold levels exist [20-22]. Further research on the fundamental origin for these will follow.

#### *ii. Semiconducting properties of surface oxide films*

The surface oxide layer of a metal substrate plays a crucial role for activity and stability at the polymer/metal interface. The Kelvin probe technique directly provides information about the work function of the surface oxide which can be interpreted also as electrode potential, which has a direct importance for the electrochemical processes occurring at the interface [7]. However, the electrode potential obtained by SKP in air does not correlate with electrochemical activity of the material immersed in an electrolyte [6]. For the delamination behaviour at the metal(oxide)/polymer interface not the band gap is important but the position of the conduction band edge which can be measured by SKP; this is further investigated by combination with UV light of defined wavelengths.

Tailoring of the surface oxide provides direct control of the stability at the polymer/metal interface [7]. One aim is to develop zinc alloy coatings which intrinsically lead to optimised surface oxide layers. One way for their preparation that is investigated is the use of ionic liquids.

Another question which is investigated is the role of zinc corrosion product layers precipitating on exposed steel surfaces. These corrosion product layers are generally of hydroxide and oxide nature. The capability to suppress oxygen reduction which is of crucial importance for the long-term corrosion performance at defects and cut-edges does vary over an astonishingly wide range and depends on a number of factors. It is our aim to provide the first fundamental knowledge on the ORR inhibition of these layers.

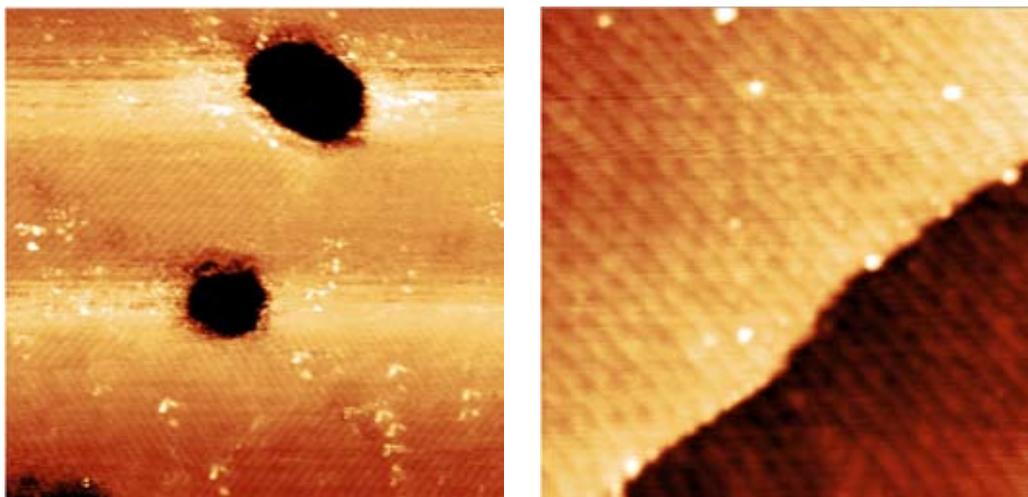
#### *iii. Wetting and interfacial reactions at metal/metal melt interfaces:*

Directly related to the last mentioned topic is the wetting behaviour of zinc (alloy) melts on steel surfaces. Here the high amount of inert surface oxides found after recrystallisation annealing of high strength steels is a considerable problem. The wetting kinetics and the nature of the resulting interfacial layer can be directly studied by a special device developed at MPIE [19]. Main factors are amount and size of oxide islands on the surface, which is tried to understand by studying the wetting behaviour on dedicated model samples, such as iron filled microporous alumina [24]. Different approaches for improving the wetting behaviour and the quality of interfacial adhesion are investigated within the frame-work of two European research projects. One focuses on novel annealing techniques, including ultra low partial pressures of hydrogen, the other on the possibility to cover the inert oxides by sol-gel applied iron oxide. Although the projects are application driven, the scientific questions addressed at MPIE are of very fundamental character. For instance, in order to understand the processes at the surface of the highly alloyed (and carbon rich) steel, it is important to know what the effective partial pressure of oxygen at the surface is. This question is not easy to investigate. One strategy at MPIE is to build up a high-temperature quartz crystal microbalance (QCM) set-up.

#### *iv. Intelligent self-healing concepts for corrosion protection*

Intelligent self-healing capabilities of organic and inorganic coatings are of central importance for the production concepts of the future. The scientific questions addressed in this topic span a wide range of research fields: from the fundamental mechanisms of corrosion inhibition to the parameters controlling particle codeposition during galvanizing quite a number of complex problems have to be understood. Currently, the main research addresses two questions: how to get an optimized performance by conducting polymer based organic coatings and particle modification for optimising incorporation during electrodeposition of zinc.

We could show that conducting polymers are suitable for intelligent corrosion protection, and that by preventing macroscopic percolation networks of the conducting polymer in the composite coating the danger of sudden coating break-down that is an inherent property of all conducting polymers [14] can be averted. This could be further substantiated. However, problems with anodic corrosion at the interface between metal and composite coating still exist. Although the buried interface underneath



**Fig. 3:** a) Left image shows a  $100 \times 100 \text{ nm}^2$  scan of the model alumina surface, showing two striped domains. b) Right image:  $40 \times 40 \text{ nm}^2$  close-up.

the conductive polymer layer should be invisible to the Kelvin probe technique, for some metals SKP was found to be directly applicable for studying the interfacial corrosion reactions in-situ. This has to do with the formation of an insulating interfacial corrosion product layer that leads to Fermi level non-alignment which can be detected by SKP. Based on this a procedure was developed for studying the interfacial corrosion kinetics.

While we could successfully study incorporation of molecules into, encapsulation of and diffusion from microporous silica particles [15,16], the incorporation of silica particles during electro-galvanising is still not understood. Our current research is addressing exactly this question.

#### v. Surface modification by organised monolayers

As mentioned above, the performance of organic monolayer films critically depends on molecular and nanoscopic defect structures, such as domain boundaries. Although some types of molecules, e.g. phosphonates, are already used in industry as adhesion promoting SAMs, still not much is known about the defect structures present in these SAMs. This is due to the fact that up to now these SAMs are only applied to aluminium alloy, which makes e.g. the application of STM impossible. Now we successfully managed to prepare an atomically smooth ultra-thin alumina model layer on niobium that is not only suitable for STM (see Fig. 3) but also survives immersion in water or aqueous electrolyte. First results on phosphonate monolayers on these model samples were obtained, but need to be verified.

#### vi. SKP as a tool for general application for processes at buried interfaces

The activities within this topic are described in the selected highlight paper "Application of SKP for *in situ*

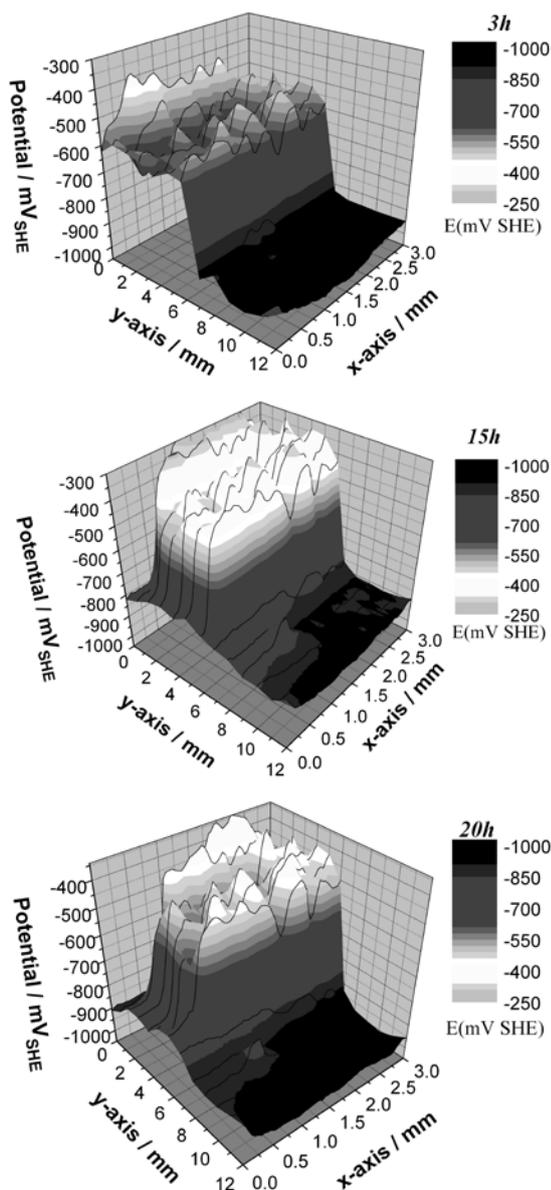
monitoring of ion migration along insulator/insulator interfaces" (page 187) and the reports on the CD lab for "Diffusion and segregation mechanisms during production of high strength steel sheet" (page 33) and on the "Novel SKP for integration into synchrotron beam line" (page 35).

#### Adhesion and Thin Films (G. Grundmeier)

In the course of the appointment of Prof. Grundmeier as a full professor at the University of Paderborn an agreement between the MPIE and Prof. Grundmeier was established that allows finishing the already started projects of his research group under his guidance at the MPIE. As a result, all projects could continue without any significant interruption. The research was covering the following topics:

- Plasma surface chemistry on oxide covered metals (zinc-alloys, aluminium)
- Structure and stability of metal and oxide nano-structures
- Barrier properties of organic coatings and defect formation during mechanical forming coated metals
- Fundamentals of molecular adhesion of organic layers on oxide covered surfaces

It was demonstrated that the surface electrode potential of ZnMg-alloys as determined by means of the atmospheric Kelvin probe measurements could be selectively shifted about some tenth of a volt in the cathodic as well as anodic direction by tailoring the surface chemistry in reducing and oxidizing plasma treatments [25]. Further, by using ultra-thin Si-organic plasma polymer layers the potential of zinc surfaces was adjusted in a way, that it became equal to the free corrosion potential of zinc in aqueous media [11]. Both techniques are well suited to reduce or even diminish the driving force for cathodic de-adhesion processes



**Fig. 4:** Delamination of a lacquer in humid atmosphere on  $MgZn_2$  after  $Ar/H_2$  (right) and subsequent oxygen (left) plasma modification measured by means of the Scanning Kelvin Probe technique.

on zinc alloys. However, mechanical deformation of specimen coated with ultra-thin plasma polymer layers led to nanoscopic defects resulting in an anodic shift of the potential and corresponding destabilization of the interface (Fig. 4).

The modification of oxide covered aluminium surfaces by means of  $H_2O$ -plasma treatments led to an increased hydroxyl density on these surfaces and thereby to a significantly increased adsorption kinetics of organophosphonic acids [12].

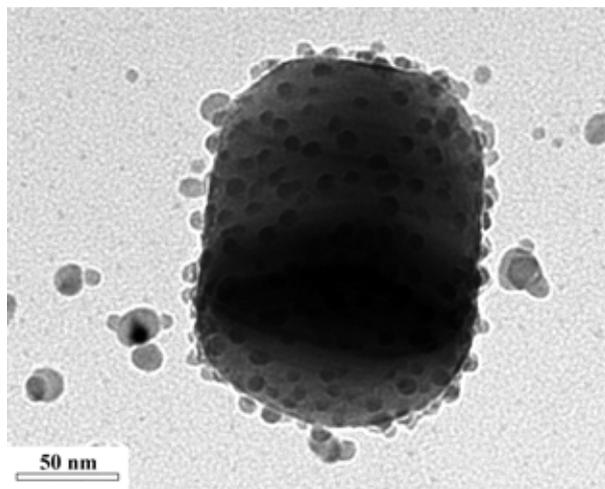
The process of electrochemical Ostwald ripening of silver nanoparticle monolayers within perfluorinated matrices could be observed. Processes on the surface on these nanoparticles within nanocomposite

systems have a significant impact on the silver release properties of antibacterial Ag/polymer-nanocomposites [13] (Fig. 5).

XANES and EXAFS investigations during the growth of Ag nanoparticles within polyelectrolyte networks were started. It was demonstrated that by exposure to light one fraction of the embedded silver is reduced from  $Ag^+$  to  $Ag^0$  even before the chemical reduction [26].

Combined experimental and theoretical investigations on the molecular adhesion of organosilanols on zinc oxide surfaces were performed in a joint venture with the atomistic modelling group. As an example the adhesion forces of amino- and silanol functional groups of a bi-functional molecule were determined [27].

The last remaining research activity of the Adhesion and Thin Films group dealing with the adhesion and delamination of water-borne lacquers on iron substrates will be finished end of 2009. With the expiration of the Christian-Doppler Laboratory for Polymer/Metal Interfaces in 2010 all research activities of Prof. Grundmeier at the MPIE will be finished.



**Fig. 5:** Electrochemical Ostwald ripening of Silver nanoparticles: TEM measurement of silver nanoparticles in a perfluorinated matrix after 12 days of exposure to an aqueous environment.

### Interface Spectroscopy (A. Erbe)

Established in November 2007, the Interface Spectroscopy group will focus on the in-situ characterization of buried interfaces in complex environments using optical, non-destructive methods. On the typical solid/ (complex) liquid interface encountered in electrochemical systems, we aim at understanding the structure and dynamics of the (complex) liquid side. Complex liquids referred to here include aqueous or other liquid electrolyte



solutions, polymers, amphiphiles, or molecules with a shape-anisotropy. In addition, the group will complement ongoing activities in the department and provide our expertise when light is needed to transform or characterize a surface.

Some of the techniques will continue as previously used in the Adhesion and Thin Films group headed by Prof. Grundmeier. As an extension of previous work, increased focus will be on quantitative work. As such, we are currently using a variety of infrared spectroscopic techniques, with the main focus on reflection techniques to gain insight into molecular composition, structure, conformation and orientation at interfaces. In collaboration with the department's simulation group (Dr. Biedermann), an important topic in the future will be the calculation of spectra given a molecular structure to aid the interpretation of experimental spectra. A very important part is the use of ellipsometry to understand surface structures. Ellipsometric techniques detect the change in polarisation of light after contact with a sample. Certain routine applications exist, e.g. in the determination of layer thicknesses of thin layers on substrates, or the determination of the optical constants (i.e. the refractive index and absorption coefficient, the dielectric function at optical frequencies) of pure, uncoated substrates. Further applications are possible with a thorough understanding and optimization of the measurement schemes. The use of Raman spectroscopy at interfaces will also continue.

Existing collaborations from previous works will continue to play a role in the new projects at the MPIE. These include collaborations with Edwin Weber (TU Bergakademie Freiberg, organic synthesis of e.g. self-assembled monolayer forming materials), Andrew Nelson (University of Leeds, UK, hanging mercury drop electrode), Patrick Kölsch (University of Heidelberg, sum-frequency generation), Lars J.C. Jeuken (University of Leeds, UK, redox-active membrane proteins), Narayan Chandra Mishra (Indian Institute of Technology Roorkee, India, tissue engineering and cell culturing), and Chiafu Chou (Academia Sinica, Taiwan, microfabrication).

Since our scientific work is at its beginning, many things here are about the future, that is projects which are about to start or have only just started. A few examples of the topics we have started to work on will be presented below.

#### *i. The infrared-transparent metal electrode*

An important problem when using optical spectroscopy to characterise thin films at a metal surface are the unfavourable boundary conditions resulting from the optical properties of metals. For an ideal

metal in the optical sense (i.e. an absorption coefficient approaching infinity, a real part of the refractive index of 0), the intensity of light directly at its interface is zero. That makes, as some textbooks put it, a metal surface the ideal place to "hide" e.g. a monolayer or organic material. We owe the success of such experiments under certain conditions to the fact that real metals behave slightly different than ideal metals. However, the characterisation of metal surfaces is more difficult than on other surfaces. The main problem in such experiments is that the surface contributions to a spectrum are damped compared to the contributions from a bulk sample, e.g. in an aqueous solution above the sample.

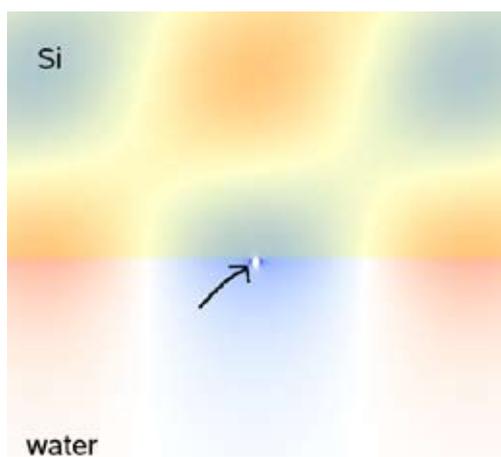
As an alternative, the use of internal reflection experiments is also difficult when metals are to be studied. In such experiments, one aims at "transporting" the light through a metal to its surface. Because of the high absorption in metals, this is actually rather difficult. Especially in the infrared wavelength range this works only for extremely thin metal films up to 20 nm thickness. In a recent work about optical tunnel barriers, Hooper et al. (Phys. Rev. Lett. 97 (2006) 053902) have shown that at visible wavelengths, a large transmission through silver layers is possible by using impedance matching interlayers between the metal and transparent medium of incidence in an internal reflection geometry.

Consequently, it is tried to use their fundamental result about the properties of optical tunnel barriers at visible wavelength and try to transform it to the infrared range. Already, conditions for the optical constants and thickness of these interlayers have been worked out in relation to the substrate, so that in a next step, a suitable system for spectroscopic applications needs to be realized.

#### *ii. Surface-enhanced infrared absorption spectroscopy (SEIRAS)*

One way of conducting sensitive spectroscopic experiments on metal surfaces is the use of small particles, which give rise to various optical effects leading to an enhancement in the spectra. While for the sister-technique surface enhanced Raman scattering, the mechanisms leading to the enhancement are well understood and a quantitative prediction of the enhancement is possible, this is not the case for SEIRAS. Theoretical models are so far limited to effective medium models. Recipes for surface preparation give random-rough surfaces, where probably only parts of the surface contribute to the spectral enhancement.

Simulations of the field distribution of the electric fields around nanoparticles on a surface using a



**Fig. 6:** Snapshot from a FDTD simulation of the electric field amplitudes around a metal nanostructure on a surface. Light with p-polarization of a vacuum wavelength of  $5\ \mu\text{m}$  is incident from the upper medium, which has a refractive index of 3.3 resembling silicon. The light is totally reflected from the interface and generates an evanescent wave in the lower medium with refractive index of 1.3 (corresponding to water). The white circle in the centre of the image represents a metal nanostructure (diameter  $\sim 20\ \text{nm}$ ). The amplitude of the electric field is represented by the blue (positive) and red (negative) colours. The dark parts near the metal particle are clearly visible and show the presence of an increased amplitude near the particle surface.

finite difference time domain (FDTD) approach have been started. An example is shown in Fig.6. When a p-polarized incident plane wave generates a longitudinal evanescent wave which interacts with a gold cylinder, there is a clear increase of the electric field near the surface of the cylinder. This effect is presumably due to the presence of a radial field in the vicinity of the metal nanostructure. A quantitative estimate of the enhancement and experimental checks are to be performed in the next stages.

### iii. Ion distribution around charged surfaces

In the past, we managed to evaluate the ion distribution around charged particles by ellipsometric light scattering and compare it with theoretical models [28]. On the other hand, there are ongoing activities in the department's Atomistic Modelling group to simulate the ion distribution around charged surfaces in the presence of a metal tip.

Since planar electrode offer a great level of control over surface potentials and hence charge densities, and reflection ellipsometry is able to characterise refractive index differences with a high sensitivity, the quantitative evaluation of an ion concentration profile near the surface seems feasible.

### iv. Electronic properties of oxide layers

The use of spectroscopic ellipsometry offers a great potential to characterise native oxide films,

because it enables a sensitive measurement of optical spectra without the need of a reference.

While for semiconductors, its application is standard practice to measure the thickness of thermally grown or native oxide layers on silicon, the application to native oxides of metals is limited so far. One notable example relevant in corrosion protection is zinc. Initial experiments have successfully determined the band gap of the native zinc oxide layer. More detailed modelling of the obtained spectra will enable a characterisation of the band structure on the whole.

Future plans include such experiments in connection with the combinatorial mixed oxide libraries prepared in the group „Electrochemistry and Corrosion“ in the department.

### v. Spectroscopic studies of delamination

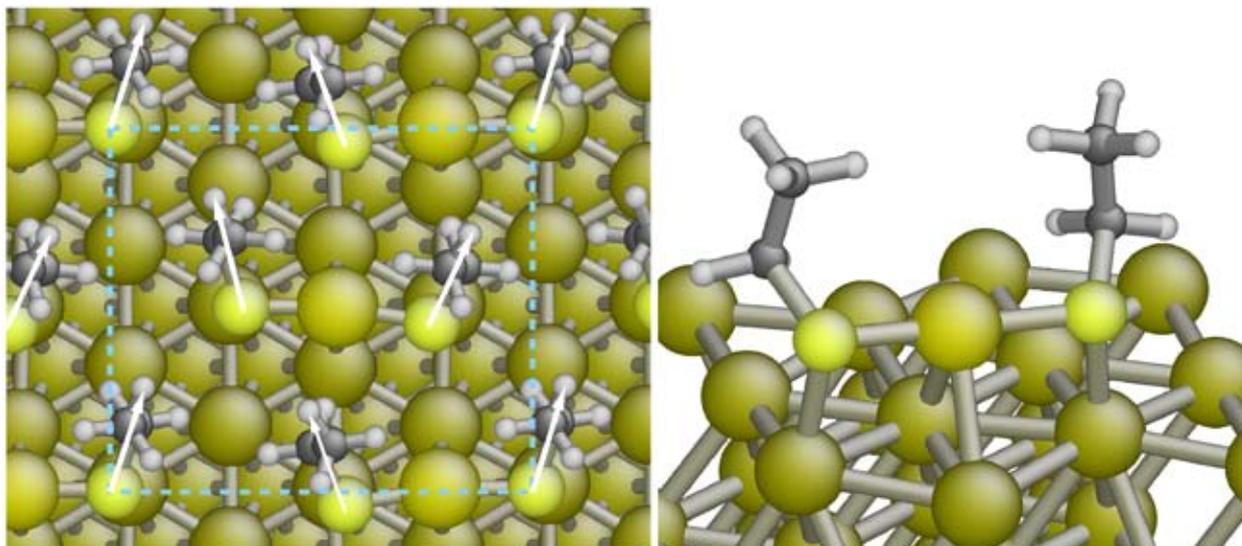
Systematic studies of delamination have a tradition within the department. We are currently working on the concurrent in-situ detection of water and ions under polymer films. There are a number of cations and anions which can be detected in the infrared (ammonium, sulphate, nitrate, phosphate, carbonate). Initial experiments in aqueous solutions of these salts have proven the feasibility of such studies.

## Atomistic Modelling in Interface Science (A.T. Blumenau / P.U. Biedermann)

The motivation of this theory group in an experimental department is to gain atomic level insight into key processes at the interfaces/surfaces, inaccessible by other means. For this purpose, a close interaction with the experimental groups is essential in order to define interesting problems, to develop a feasible theoretical approach, and to validate the conclusions. The focus is on the atomistic structure in adhesion/de-adhesion processes and electrochemical reaction mechanisms.

First-principles electronic structure methods are applied to develop a realistic atomistic structure model and to understand the binding interactions and reactivities. For larger and more complex problems, higher scale methods are used as, e.g., statistical thermodynamics and force-field or FEM simulations. The ab initio electronic structure approach is common to the Computational Materials Department with which a close exchange of ideas exists and the computational infrastructure is shared. However, here the focus clearly is on surfaces and chemical reactions.

One major line of studies focuses on the adsorption and de-adhesion of typical adsorption promoters



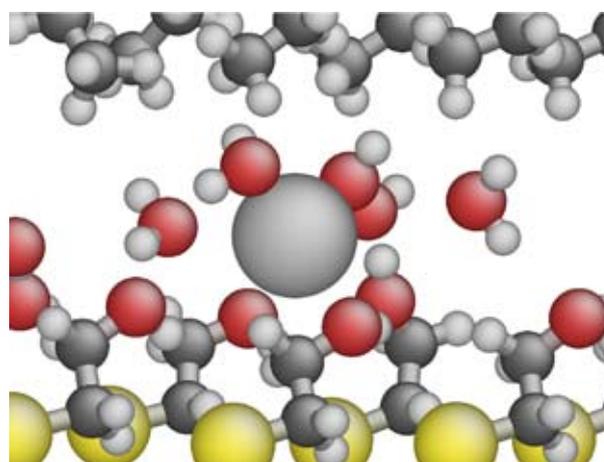
**Fig. 7:** New  $c(4 \times 2)$  structure of the ethyl thiol/Au(111) SAM with Et-Au-Et moieties binding on top of surface gold atoms in agreement with results from diffraction experiment.

such as silanols and phosphonates on the oxide or hydroxide surfaces of aluminum and zinc. Hydrogen-bonding has been compared to covalently bound structures to decipher the binding mechanism on the various substrates. On these surfaces, the competition for binding sites with water molecules always present at ambient conditions plays an important role. Therefore water adsorption was also studied and kinetic Monte Carlo simulations of temperature programmed desorption were used for validation with experiment.

The polar zinc oxide surface ZnO (0001) shows various stabilization mechanisms in the presence of oxygen and/or water. Amorphous ZnO surfaces crystallize in strongly alkaline electrolytes. Annealing of these substrates allows to grow large atomically flat terraces. Complementing these experimental findings, a theoretical surface phase diagram was developed from DFT calculations, identifying the most stable surface structures as a function of temperature and oxygen/water partial pressure in excellent agreement with experimental results.

Self-assembled monolayers (SAMs) of thiols on gold show a covalent bonding mechanism. Their strongly inhibiting properties for electrochemical reactions, as, e.g., the oxygen reduction reaction (ORR) are studied as a function of molecular structure and film organization in the group of Dr. Rohwerder. In a parallel DFT study, a new, more stable SAM structure involving a reconstruction due to gold adatoms was developed, that may reconcile the binding site controversy and offers an elegant explanation for the formation of vacancy islands. In the thiol-Au-thiol moieties shown in Fig. 7, the sulfur head groups form one bond to the adatom and one to the surface gold atom below, and hence are close to the on-top position found in photoelectron diffraction and grazing incidence X-ray diffraction studies.

Thiol/gold SAMs also allow preparation of interfaces with controlled structure, composition and properties. SKP-studies of ion migration along the interface of a model coating (PVB or paraffin) with a structurally well characterized SAM of varying hydrophilicity were taken as starting point to develop methods to simulate ion incorporation and migration at an interface (Fig. 8). A strategic collaboration with Dr. Nico van der Vegt from the Max Planck Institute for Polymer Research and the ICAMS will extend these approaches to realistic models of the interphase at polymer/metal (oxide) interfaces. The multi-scale approach will include DFT studies of the binding interactions between the surface and polymer, development of a coarse-grained force-field to describe the polymer equilibration over long time spans and reintroducing atomistic detail to study diffusion. The facile migration of ions along or near the coating/metal interface leads to galvanic coupling in anodic and cathodic delamination.



**Fig. 8:** Snapshot from a MD simulation of diffusion in a paraffin/mercaptoethanol/Au(111) SAM interface.

The electrochemical oxygen reduction reaction (ORR) is one of the driving forces of metal corrosion. It also plays an important role as cathodic reaction in fuel cells. The ORR has a complex multi-step mechanism with reactive intermediates that may lead to a chemical degradation of coatings in cathodic delamination. The reaction mechanism of ORR in aqueous electrolyte has been decomposed into elemental steps of electron or proton transfers yielding a comprehensive set of intermediates. Among these, OH radicals were found to have the highest reactivity in hydrogen abstraction reactions. The relative stability of several intermediates changes with the reaction conditions, indicating that the sequence of reactions along the minimum energy reaction path changes with pH and electrode potential. Spectroscopic detection of these intermediates is planned in collaboration with Dr. Erbe. The work will be extended to include the electrocatalytic effect of metal electrodes, as well as other important reactions including the hydrogen evolution reaction (HER) and metal dissolution.

The local electric potential, electric field and ion concentrations are essential parameters determining electrochemical reactions. The long-range interaction of electric potentials and ion concentrations in the diffuse double layer may be efficiently modeled by the finite element method (FEM). As a first step in this direction, perturbations of the electric double layer by the probe electrode in scanning electrochemical potential microscopy (SECPM) are analysed to develop a combined experimental/theoretical access to double layer properties. Future multi-scale simulations will combine local information on specific adsorption and electrochemical reactions from DFT calculations with long-range simulations of concentration, potential distributions and material transport at the FEM level.

### Electrochemistry and Corrosion (A.W. Hassel)

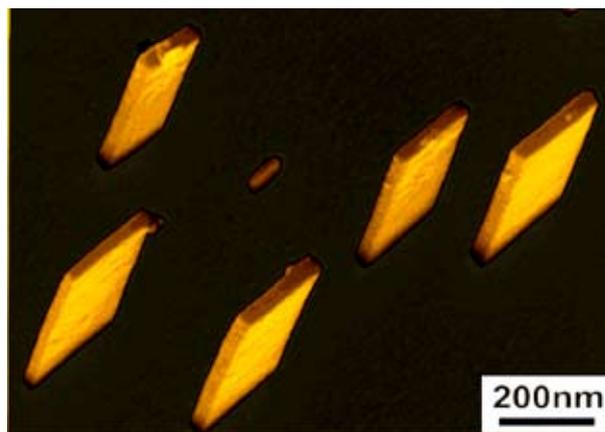
The Electrochemistry and Corrosion group is still working on chemical and engineering aspects of materials with a focus on their microstructure, crystallography and reactivity. A thorough understanding of the reaction mechanism e.g. passivity is not only used to predict materials stability but rather employ such processes for a design on a nanoscopic and even atomic level.

Major research areas are:

- i. Nanotechnology: metallic nanowires and nano-devices
- ii. Corrosion Science: from corrosion to chemical mechanical planarization (CMP)
- iii. Passive films: from understanding the mechanism to design rules
- iv. Combinatorial material development: accelerating the search for new materials

### i. Nanotechnology: a link between directional solidification and electrochemistry

A generalized method has been established that allows employing eutectic systems to produce nanostructures such as wires, wire arrays, pore arrays and even more complicated structures. This approach has been systematically extended in terms of systems, physics, modification and applications. The approach is now also applicable to produce Mo nanowire arrays from the ternary-quasibinary NiAl-Mo system [29] and Cu wires from the Ag-Cu system [30]. The preparation of Au nanowires turned out to be more complicated since no binary phase diagram is known in which Au forms the minor phase of a eutectic system. However, the Fe-Au is interesting since it includes a eutectoid transformation which was successfully used to produce iso-oriented Au nanobelts [31] which are shown in Fig. 9. Further extension of this method can be expected e.g. for the processing of eutectics in which at least one of the elements is not a metal. Si nanostructures have been produced from a directionally solidified Al-Si eutectic [32]. This material is now considered as an electrode material in a Si-air battery.



**Fig. 9:** Iso-oriented Au nanobelts. A eutectoid Fe-Au alloy was directionally processed to allow a controlled phase separation. Parts of the Au nanobelts are finally released under electrochemical control. The Au nanobelts are single crystals with an unusual 110 surface and show identical crystallographic orientation in all three directions.

Directionally solidified material was used as a model system for the quantitative investigation of the Lotus effect [33] in collaboration with Prof. Adelung and Prof. Faupel from the university in Kiel. Mechanical investigations on single nanowires were performed by means of an AFM mounted in a SEM to determine anisotropy of the yield stress [34] in another collaboration with Dr. Philippe and Prof. Michler. A single crystalline W nanowire was employed to build a NEMS within a multi-institute initiative, which is not only an impressive technical advancement but also delivers physical properties of these structures [35]. Further work was conducted to determine quantitatively the growth parameters of systems



such as the NiAl-W [36] and a detailed description of the materials structure [37] in collaboration with the group of Dr. M. Rohwerder and the National Institute for Materials Science in Tsukuba, Japan which is a designated partner institute of the MPIE.

Selective electrodeposition of Au onto the tip of Re nanowires was used as a method for the modification and a possible later functionalisation [38]. The mechanism itself was studied to understand the kinetic hindrance in the initial steps of electrodeposition due to the required reduction of the rhenium oxides prior to electrodeposition [39]. Based on these results a modification of W nanowires was finally also successful [40].

A smart anodising procedure allowed producing an extremely small pH-sensor based on a single W nanowire. After electrochemical tapering of a millimetre sized sample, a single nanowire was released from the very tip by selective matrix dissolution. The oxide film on the W nanowire was subsequently modified to become pH sensitive [8]. Such a sensor with a diameter of only 100 nm and an aspect ratio of more than 100 can be a useful tool for measuring the pH value in corrosion pits or in a single biological cell. Our approach for the production of nanowires was always considered as a realistic production process for metallic nanowires. We have therefore performed a batch to produce 1 g of nanowires. This may not sound very much, but for nanowire segments with a diameter of 100 nm and a length of 300  $\mu\text{m}$ , 1g corresponds to  $5 \cdot 10^9$  nanowires [41], one for each human in the world! A similar approach is being made with Au nanobelts, but in this case already smaller amounts may be useful e.g. for biosensors in which the Au nanobelts are chemically modified [42].

This research aims at developing a generalized set of strategies for producing, modifying and employing metal nanostructures including novel properties resulting from the inherent structure of these materials.

#### *ii. Corrosion Science: from corrosion to CMP*

The group's activities in the field of corrosion are directed towards an understanding of corrosion mechanisms. This knowledge may be later used to develop corrosion protection strategies on one hand or in employing the corrosion in a useful way.

A novel experimental setup has been constructed that allows studying single particle impacts in multi-phase flow induced corrosion [43]. It allows studying the effect of particle morphology and a quantitative description of the normal (indent) and lateral (scratch) component during impact under various angles [44]. This method can also be used to explore the

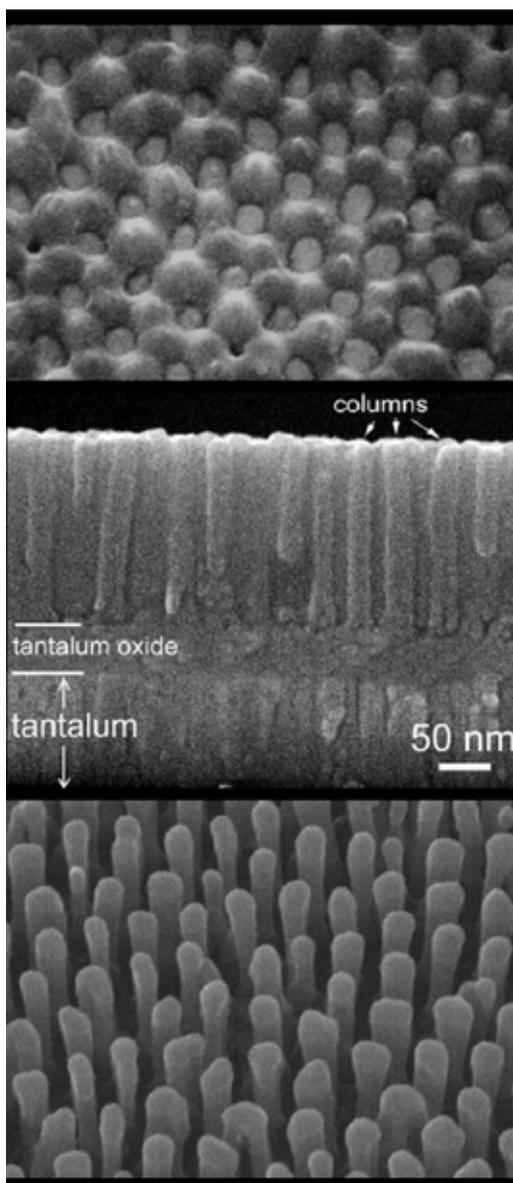
reaction mechanism in CMP, an important industrial process in the semiconductor industry in the copper damascene process. The adsorption kinetics and protection efficiency of alternative inhibitors such as sorbate has been studied in collaboration with Prof. Y. Ein-Eli from the Technion, Israel. With its unique time resolution of only 1 microsecond it was possible to determine quantitatively the adsorption kinetics of sorbate and its efficiency for extending the range of passivity of copper [44].

New steels such as the ferritic FeAlCr steel, which show a good deep drawability, were developed in the Department of Metallurgy at the MPIE. This material is not only a promising candidate for automotive applications but is also an interesting model material for studying orientation dependent corrosion and passivation properties. Reactivity imaging of this material was performed by means of scanning electrochemical microscopy, demonstrating that each grain shows the same reactivity in the passive state [5]. The grain boundaries on the other side show a higher activity and differences between the various orientations were found in particular under active corrosion conditions. A quantitative description of the anisotropy of dissolution was derived from a combination of electrochemical measurement, orientation imaging and topography measurements.

Progress in corrosion science is often connected with the development of new techniques or with the improvement of existing ones. A universal alternating immersion simulator for accelerated cyclic corrosion tests has been developed which is capable of simulating the formation of protective rust layers on weathering steels [4]. This device can also be used to study active passive transitions in cyclic corrosion test or to simulate the attack of biomaterials in artificial saliva.

#### *iii. Passive films: from understanding the mechanism to design rules*

Passivity of metals and alloys is still an important issue for predicting the stability of a metal or an alloy. Understanding the growth mechanism and the transport processes in passive films is a solid basis for a further improvement or a desired modification. The anodising conditions during the growth of thin barrier oxide films on pure aluminium will strongly influence the trap distribution in the oxide, which will in turn have a strong influence on the electronic transport processes [45]. The deposition of Au nanoparticles on thin aluminium oxide films and its subsequent embedding by further oxide growth is part of an ongoing research initiative in collaboration with Dr. Rohwerder to prepare surfaces with a designed resonance tunnelling structure or electrocatalytic property [46].



**Fig. 10:** Smart anodising of an Al-Ta film allows forming honeycomb structured anodic aluminium oxide that is filled by Ta oxide pillars growing into the pores (a). A side view shows the oxide covered Ta metal film which carries the nanostructured oxide ensemble (b). Selective chemical dissolution releases the Ta oxide nanopillar array (c).

Several aspects of the surface processing of shape memory alloys from the NiTi class have been addressed. The mechanism of electropolishing has been investigated by means of a rotating disc electrode, and it was demonstrated that it follows a salt film mechanism [2]. Also the ternary NiTiCu alloy can be electropolished in a methanolic sulfuric acid [47]. The high dissolution rate equilibrates the surface composition for both materials to the bulk composition and simultaneously yielding a perfect surface finish of the material. Various surface modification methods have been employed for surface engineering of NiTi. Each of these methods aims at creating a virtually

Ni free or at least significantly Ni depleted surface. At the same time a hard and wear resistant surface is desired. High voltage pulse anodising is one way to produce thick oxide layers during the resulting surface plasma reaction [3]. Other strategies involve high temperature reactions for oxochloridation [1] or for reduction annealing [48]. Both strategies yield hard coatings with lower Ni content, but the mechanical properties and the corrosion resistance still have to be explored.

The term smart anodising is quite appropriate for an electrochemical route that produces nanopillar arrays. A sandwich Al/Ta or Al/W layer is oxidised to form a regular array of nanopores in the aluminium oxide which is forming during anodisation. The underlying Ta [49] or W [50] grows its oxide into these holes before the aluminium oxide is removed (Fig. 10). Such structures may be used for a surface functionalisation or with its uniform spacers for the production of displays.

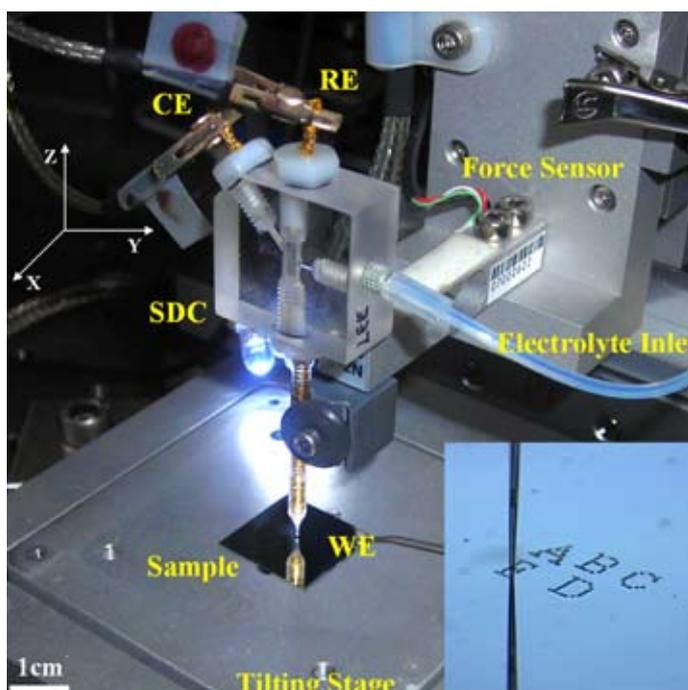
*iv. Combinatorial material development: accelerating the search for new materials*

A major progress has been achieved by setting up a fully automated scanning droplet cell (Fig. 11). This versatile electrochemical method allows performing all electrochemical methods in a small confined zone [51]. This electrochemical high throughput method can be applied to composition spread samples which are prepared by evaporation of different metals from 2 or 3 sources onto a substrate. The resulting alloy library is first characterised in terms of composition (EDX) and microstructure (SEM and XRD) before a comprehensive set of electrochemical experiments is performed at each position that is, for each composition. Subsequent stepwise anodisation alternating with electrochemical impedance spectroscopy (EIS) yields a complete set of oxide formation parameters as well as the specific resistance and the dielectric permittivity for each local alloy. Potential dependent EIS for a given oxide provides the semiconductor properties such as flat band potential and donor density as a function of the composition. This method has first been applied to Al-Fe alloys. An unexpected singularity was found for a content of 12 % Fe. This was attributed to a percolation threshold within the oxide [10].

Binary and ternary valve metal libraries including the elements Hf, Nb, Ta and Ti are being investigated in collaboration with Prof. A. Wieck from Ruhr-Universität Bochum and Prof. A. Ludwig from caesar Bonn. Ta-Ti alloys are of interest for developing high k materials [52] for the semiconductor or condenser industry. Ti-Hf [53] and Ti-Nb [54] on the other hand are promising candidates for  $\beta$ -Ti alloys, a mechanically favourable biomaterial. For each of



these materials a wide range of composition was investigated with a resolution of 0.5% in composition. The information obtained from each single library exceeds by far the sum of all information published so far on the electrochemical properties of each alloy. Further alloys are now being investigated and other material classes will be investigated including the ternary Al-Mg-Zn system for the development of advanced corrosion protection coatings.



**Fig. 11:** Photograph of the scanning droplet cell which has been developed into a versatile tool for studying local electrochemistry in corrosion science as well as high throughput screening in combinatorial alloy development.

### Interface Structures and High Temperature Reactions (F.U. Renner)

The research activities of the new group focus on reactions and structures of interfaces at elevated and high temperatures. We study mainly model alloy systems but also commercial steel grades. These samples are examined in well-defined gas atmospheres, including ultra high vacuum (UHV) as well as corrosive gases. In the centre of the group are in-situ studies employing intense X-ray beams, which are provided at synchrotron radiation sources. These in-situ studies include buried solid-solid and solid-gas interfaces as well as solid-liquid and electrochemical (solid-electrolyte) interfaces.

The group is currently building up new experimental equipment for the UHV preparation of single crystal surfaces and thin epitaxial films, and the possibility to directly transfer the prepared samples into special reaction chambers. This frame will provide as well in-situ diffraction chambers for reactions in gas as well as liquid environment [55] which can be detached to be used for synchrotron radiation studies. The new equipment will add to the experimental possibilities that have been recently built up within the former High-Temperature Reactions group by PD Dr. Michael Spiegel and coworkers.

The knowledge of atomic structures and the understanding of atomistic reaction mechanisms are a prerequisite for furthering corrosion protection but are also urgently needed for controlling reactions in general in a broad variety of technological fields as for example in fuel cell catalysis or in battery technology.

In this sense the research of the group is dedicated to employing synchrotron radiation and laboratory-based techniques to corrosion processes and structural changes in a broad sense: initial scale growth, electro-deposition and dissolution, dealloying, ion intercalation, and so on.

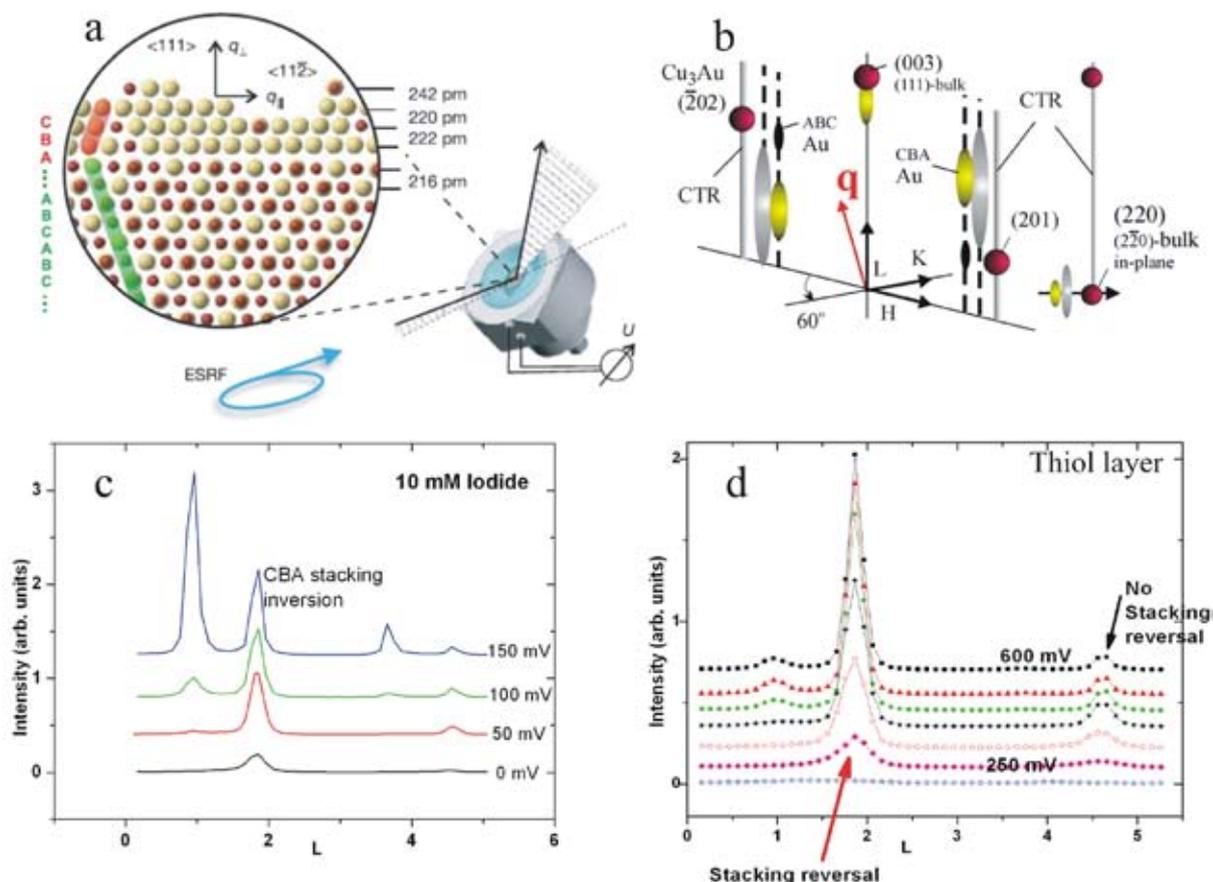
The main current and future activities include:

1. In-situ measurements employing synchrotron radiation techniques for studying corrosion and corrosion-related topics
2. Exploring the interaction of defined gas-atmospheres with metallic model systems at elevated and high temperatures
3. Studying elemental, binary, and multi-element model systems in corrosive surroundings

The current projects of corrosion and corrosion-related studies include:

#### *i. Initial oxidation of binary and ternary alloys*

Metallic model systems and in particular single crystal surfaces and epitaxial (textured) thin films provide a means to elucidate detailed mechanisms of oxide scale formation. The influence of parameters like density and nature of grain boundaries on the formation and stability of oxide scales will be studied in detail. Another topic will be surface modifications and the application of thin oxide films to influence and control the further oxide scale growth. As a first project the initial and further oxidation of clean and surface-modified NiAl and FeAl single crystal surfaces [56,57] will be studied in collaboration with Dr. Andreas Stierle (Max-Planck-Institut für Metallforschung, Stuttgart).



**Fig. 12:** In-situ grazing incidence X-ray diffraction reveals structural details of the surface of  $\text{Cu}_3\text{Au}(111)$  at controlled potential in  $0.1 \text{ M H}_2\text{SO}_4$ . (a) Experimental setup and the initial passive surface with a top layer of stacking-reversed Au-rich alloy. (b) Sketch of the reciprocal space indicating the position of the Bragg intensity of the initial reversed ultrathin Au-rich film, of subsequently emerging thicker Au islands, and of the finally occurring substrate oriented Au layer as revealed in earlier studies. The L-scans ( $20L$ , in the used surface unit cell) along the features stemming from the passive surface layer in an iodide containing electrolyte (c) and with a thiol modified sample (d) show the large retarding effect of the thiols.

### ii. High temperature corrosion and short term annealing

Activities to study the influence of well-defined gas atmospheres in the context of high-temperature corrosion and short term annealing will be partially continued. These studies benefit from the equipment and expertise of the former High-Temperature Reactions group. Special emphasis is put on building up further in-situ chambers and a better connection to UHV analysis equipment.

### iii. In-situ studies on dealloying of binary alloys

Dealloying or selective dissolution can take place in an electrochemical environment for alloys of elements with a large difference in Nernst potential. Dealloying plays a role in stress corrosion cracking of steel. On the other side dealloying can produce also interesting nano-porous films of noble metals with applications in catalysis or other fields. This phase separation mechanism is also of large fundamental interest [58]. In a continuation of earlier work [9, 59, 60] the dealloying of Cu-Au alloys forms a first topic. In particular the influence of additives to the electrolyte and of organic adlayers and polymer films is examined (Fig. 12). This work is performed

in collaboration with the group of Dr. Michael Rohwerder. A second topic will be the dealloying of Cu-Pd [61] and Cu-Pt alloys which will be studied in collaboration with Prof. Peter Strasser (Technische Universität Berlin). Further projects involving electrochemistry are envisioned in collaboration with the "Electrochemistry and Corrosion" group (Dr. Achim Walter Hassel).

### iv. In-situ electrodeposition from ionic liquids

In collaboration with the group of Dr. Michael Rohwerder we performed an in-situ diffraction study on the electrodeposition of Zn using, among others, single crystal model substrates (see also Pareek et al. in this report).

### v. Li ion battery electrodes

Many electrochemical processes which are important in corrosion (deposition, dissolution, intercalation, dealloying) are also relevant in technology, for example in batteries. Energy storage is one of the most urgent technological problems of the new millennium. In continuation of earlier work [62] structural changes during charging and discharging of Li ion battery model anodes will be further studied.



## Outlook

In the last two years the department saw major restructuring of its groups: the heads of three groups left the department and started new careers either in industry or in academics. Dr. Spiegel took over a leading research position at Salzgitter Mannesmann Research and Dr.-Ing. Grundmeier a position as professor at the University of Paderborn and a position as department head at the BAM in Berlin. In autumn 2008 also the head of the simulation group, Dr. Blumenau, left for a high level industrial position. The group is currently led by his long-time co-worker Dr. Biedermann. The leave of the former group heads left vacancies in expertise and leadership and also a certain drop in the number of projects. These vacancies are now started to be filled by the two new heads of research groups who also bring with them new valuable expertise that is now just starting to fuel new cooperations in the department. Nevertheless, at the same time the department was successful in holding its position as a well established organization, and is internationally highly visible and has strong links to the German and international steel industry. The department is internationally leading in the area of steel surface technology and hosts

many large international projects with scientific and industrial groups.

The foreseeable future is determined by the following developments:

- (1) The new theory department for Materials Science at the Ruhr-Universität Bochum (Interdisciplinary Centre for **A**dvanced **M**aterials **S**imulation, ICAMS) has officially been opened 2008, sponsored by German materials industry. It is expected, that subjects crucial for the department will be at the focus of cooperative research with the simulation group of the department.
- (2) An electrochemical centre will be founded in cooperation with the RUB, where it will be officially centred. This will enable the department to gather the critical mass for tackling crucial fundamental questions of general importance, such as kinetics of oxygen reduction or hydrogen formation on complex surfaces.
- (3) In addition a novel electrochemistry group will be founded at MPIE.

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## Research Projects in Progress

### Adhesion and Thin Films

*Fink, Grundmeier:* Analysis of thin hybrid conversion films on galvanised steel sheet

*Itani, Grundmeier:* Synthesis and analysis of metal nanoclusters in polyelectrolyte thin films

*Keil, Grundmeier:* Application of NEXAFS to the analysis of metal/polymer nanocomposite films

*Özkaya, Grundmeier:* Molecular adhesion on polyelectrolyte/silicon oxide interfaces

*Posner, Grundmeier:* Spectroelectrochemical studies of polymer/metal interfaces

*Posner, Grundmeier:* SKP studies of the mobility of hydrated ions in polymeric interfaces

*Valtiner, Grundmeier:* Single Molecule Adhesion on Zn-oxide single crystals

*Wang, Grundmeier:* Chemical Force Microscopy on oxide covered metals in corrosive environments

*Zuo, Grundmeier:* Analysis of Ag nanoclusters in TiO<sub>2</sub>-matrix for photochromic applications

### Atomistic Modelling in Interface Science

*Biedermann, Rohwerder:* Ab initio modelling of oxygen reduction in aqueous solution

*Biedermann:* DFT study of electrochemical oxygen reduction on metal and metal oxide surfaces

*Biedermann, Rohwerder:* DFT simulation of ion diffusion in paraffin/thiol-SAM/gold sandwich interfaces

*Fujita (Exeter), Blumenau:* Modelling dislocation cores and core-impurity interaction in semiconducting materials (collaboration N. Fujita, T.A.G. Eberlein and R. Jones (Exeter, UK) and S. Öberg (Luleå, Sweden))

*Hamou, Rohwerder, Biedermann:* FEM simulation of potential profile, ion diffusion/migration and electrochemical reactions at metal electrolyte interfaces

*N.N., Rohwerder, Biedermann:* Polymer structure at interfaces and ion diffusion mechanisms (collaboration with N. van der Vegt (MPI-P Mainz) and R. Drautz (ICAMS))

*Özcan, Grundmeier, Biedermann:* DFT analysis of the adsorption and adhesion of silanols on Zn-oxide single crystals

*Torres, Biedermann:* DFT modelling of alkyl-thiol self-assembled monolayers (SAMs) on gold (111)

*Valtiner, Grundmeier, Biedermann:* DFT study of silanol binding modes on aluminium oxyhydroxides

### Electrochemistry and Corrosion

*Adelung, Milenkovic, Hassel:* Extremophobic Surface structures

*Bruder, Diesing, Hassel:* Deep Level Transient Spectroscopy of anodic oxides

*Bruder, Hassel:* New approaches in electrolytic cleaning of cold rolled steel sheets

*Chen, Schuhmann, Hassel:* Bioanalytics with chemically modified nanowires

*Chen, Tian, Hassel:* Random versus organised one dimensional gold nanoparticles and their spectroscopic characterisation

*Diesing, Bruder, Hassel:* Electronic tunneling in metal-insulator-metal contacts

*Fenster, Abts, Hassel:* Chemical sensing with single nanowires

*Fenster, Rohwerder, Hassel:* Reactivity of zinc and novel zinc alloys under pH, CO and CO<sub>2</sub> influence monitored by impedance titration

*Frankel, Smith, Hassel:* Functional microstructures by selective electrochemical dissolution of a directionally solidified eutectics

*Hassel:* Combinatorial electrochemistry for the development of improved Ni based alloys

*Klemm, Hassel:* Combinatorial material development of ternary Al-Mg-Zn alloys

*Lill, Rablbauer, Hassel:* Corrosion of ferritic FeAlCr light weight steels

*Mardare, Mardare, Ispas, Hassel:* Ta coatings from ionic liquids

*Mardare, Wieck, Hassel:* High throughput screening of band gap engineered materials

*Mardare, Wieck, Ludwig, Savan, Hassel:* High throughput screening in combinatorial alloy development

*Merzlikin, Mingers, Hassel:* Hydrogen determination in coated steels

*Merzlikin, Mingers, Hassel:* Experimental determination of the diffusion coefficients in high strength steels

*Milenkovic, Hassel:* Production of nanowire arrays through directional solidification and their application

*Milenkovic, Hassel:* Gold nanostructures by directional solid state decomposition of Fe-Au alloys

*Milenkovic, Hassel:* Application of Directionally Solidified Nanowire Arrays

*Mingers, Hassel:* Delamination of thermal spray coatings

*Mozalev, Hassel:* Nanostructured aluminium tantalum composite oxides

*Neelakantan, Drensler, Eggeler, Hassel:* Electro-polishing of NiTi and NiTiCu based shape memory alloys

*Neelakantan, Eggeler, Hassel:* Microstructural aspects of passivity and corrosion of NiTi

*Philippe, Milenkovic, Hassel:* Mechanical investigations on nanowires

*Sanders, Hassel:* Metal corrosion in contact with clay

*Schmidt, Havenith, Hassel:* Terahertz Spectroscopy and Scanning Nearfield Infrared Microscopy on metallic nanowires

*Smith, Ein-Eli, Hassel:* Sorbate solutions in chemical mechanical planarization

*Smith, Hassel:* Detection of single impacts in particle induced flow corrosion

*Venzlaff, Widdel, Stratmann, Hassel:* Microbiologically influenced corrosion of iron by sulfate reducing bacteria

*Woldemedhin, Raabe, Hassel:* Surface reactivity of  $\beta$ -Ti alloys

### Interface Structures and High-Temperature Reactions

*Ankah, Pareek, Hümann, Rohwerder, Renner:* In-situ diffraction studies on dealloying of CuAu alloys.

*Borodin, Vlad, Stierle, Rohwerder, Renner:* Structural analysis of an ultrathin epitaxial Alumina layer on Nb.

*Hotar, Vogel, Renner, Palm:* High temperature corrosion of iron aluminides in humid atmosphere

*Pareek, Torres, Biedermann, Renner:* Structure of thiol and selenide adlayers on Au.

*Pareek, Borissov, Vogel, Rohwerder, Renner:* In-situ diffraction studies on Zn deposition from ionic liquids

### Molecular Structure and Surface Modification

*Ankah, Rohwerder (in cooperation with Renner and Prof. Eggeler, RUB):* Combined EC-STM and TEM investigation of the de-alloying behaviour of copper-gold intermetallic phases.

*Borissov, Rohwerder:* Fundamental investigations on the wetting and interfacial reaction behaviour at the interface liquid zinc/steel: preparation of dedicated model samples

*Borissov, Rohwerder:* Ionic Liquids for zinc alloy deposition

*Borissov, Rohwerder (in cooperation with Renner):* Interfacial reactions during electro-deposition from ionic liquids

*Borodin, Rohwerder:* MBE-based preparation of samples with well characterizable oxide surface layers as model systems for the study of surface modification and delamination

*Evers, Rohwerder:* Investigation of hydrogen uptake kinetics during wet surface treatment

*Fenster, Rohwerder (in cooperation with Hassel):* Passive zinc at high alkaline pH

*Isik-Uppenkamp, NN., Rohwerder:* Intelligent corrosion protection based on conducting polymers/

*Isik-Uppenkamp, Salgin, Rohwerder:* Mobility along interfaces

*Khan, Rohwerder (in cooperation with Dr. F. Marlow, MPIK):* Intelligent corrosion protection by nanocapsules incorporated to the zinc coating: understanding co-deposition

*Krieg, Borissov, Rohwerder:* Investigation of cathodic self-healing at cut-edge

*Laaboudi, Rohwerder:* Correlation between molecular structure and degradation behaviour for thiol monolayer films on gold as a model system for delamination at polymer/metal interfaces

*Muglali, Rohwerder (in cooperation with Prof. Wöll, RUB):* Oxygen reduction at Self-Assembled Monolayer / Gold interfaces

*Senöz, Rohwerder (in cooperation with Prof. Schuhmann, RUB):* Combining SECM and SKPFM for the investigation of localised corrosion

*Salgin, Rohwerder:* Fundamental investigation of the role of native oxide on delamination

*Salgin, Vogel, Rohwerder:* Integration of SKP as online analytical tool into Synchrotron beamline

*Swaminathan, Hüning:* Investigation of hydrogen uptake kinetics during recrystallisation annealing

*Swaminathan, Hüning:* Segregation and oxidation of alloys

*Swaminathan, Hüning, Borodin:* Fundamental investigation of grain boundary oxidation

### Interface Spectroscopy

*Ebbinghaus, Borissov, Biedermann, Rohwerder, Erbe:* Spectroscopic characterisation of metal complexes in ionic liquid

*Ebbinghaus, Erbe:* Chelatisation of trace metal compounds through amino acids.

*N.N., Biedermann, Erbe:* Detection of intermediates in the oxygen reduction reaction

*N.N., Erbe (in cooperation with Rohwerder):* Mobility of water and ions along polymer/metal interfaces

*Reithmeier, Erbe:* Transparent metal electrodes

*Vasan, Erbe:* Design of a "rough model interface"

*Yöndem, Richter, Erbe:* Diffusion of salt solutions through and under polymer films



# Department of Material Diagnostics and Steel Technology

A.R. Pyzalla

## Introduction

The department Material Diagnostics and Steel Technology focuses on the development of new steels and iron base alloys, the characterization and optimisation of dissimilar welds of steels to

lightweight materials and the development of novel techniques for material diagnostics using synchrotron radiation.

## Recruitments of New Groups

Recruitment of excellent group leaders was one main focus of building up the department "Material Diagnostics and Steel Technology" in the years 2006 and 2007. Already in February 2006 Dr. Aleksander Kostka joined the department as first of the group leaders. He became responsible for the group "*Microstructure Characterization*" where he concentrated on the determination of microstructure-property relationships in welds between steels and dissimilar materials. His tasks further included the choice, ordering and commissioning of equipment for scanning electron microscopy, a microprobe and in particular for a new 200 keV transmission electron microscope with energy filter.

In March 2006, Dr.-Ing. Haroldo Pinto, former group leader at the Institute of Material Science and Technology at TU Wien, Austria, joined the department becoming head of the group "*Material Testing*". His group aims at developing novel in-situ devices and methods for determining stresses and strains in oxide layers on Fe-base materials and Fe-Al intermetallics and residual stress analyses in welds. Dr. Pinto further has been responsible for operating the various X-ray laboratories among them special facilities for phase and residual stress analyses. Together with his co-worker Dipl.-Ing. Benjamin Breitbach, Dr. Pinto served as Radiation Protection Officer for the whole MPIE.

In February 2007, Dr.-Ing. Sebastian Weber, former group leader in the department "Material Technology"

of the Institute of Materials, Ruhr-Universität Bochum, became head of the group "*Metallurgy*". His group focuses on the development of advanced steels and wear resistant Fe-base alloys. He has been responsible for the co-ordination of smelting metallurgy services within the institute and for various customers from other research institutes, universities and industry. Dr. Weber and the technicians in the smelting metallurgy group have continued keeping the smelting metallurgy equipment at the highest standard. One of the highlights was the re-building and re-commissioning of the pressure furnace after the renovation of the experimental hall.

In spring and summer 2007, respectively, Dr. José Luis Garcia and Prof. Dr. András Borbély joined the department. Dr. Garcia, former head of the R&D department of Boehlerit GmbH & CoKG in Austria, joined the department to head the group "*Engineering Thermodynamics*", which aimed at developing new creep resistant steels. He thus has been in charge of re-establishing close contact to the Thermo-Calc developers and the community working in Engineering Thermodynamics.

Prof. András Borbély is head of the group "*Synchrotron and Neutron Methods*". His group focuses on developing new methods for the in-situ visualisation of creep damage evolution in steels and model materials.

## Infrastructure

The main contributions of the department towards infrastructure provision within MPIE were:

- alloy preparation by smelting metallurgy (Dr. Weber)
- X-ray diffraction laboratory and "Radiation Protection Officer" (Dr. Pinto)

- transmission electron microscopy laboratory (Dr. Kostka)
- co-ordination access to Thermo-Calc and Dictra Software for simulation of phase diagrams and diffusion processes in steels (Dr. Garcia)
- co-ordination of the institute library (Dr. Garcia)

## Participation in Working Groups and Committees

Members of the department joined the appropriate working groups of the Steel Institute VDEh as well as working groups of scientific societies in their field:

- working group "Electron Microscopy" (Dr. Barbatti)
- subcommittee "Alloyed Mild Steels" (Dr. Weber)
- subcommittee "Metallography" (Dr. Barbatti)
- subcommittee "Tool Steels" (Dr. Weber)
- working group "Heat-Resistant Steels" (Dr. Garcia)
- working group "High-Temperature Materials" (Dr. Pinto)
- subcommittee "Non-destructive Testing and Measurement Techniques" (Dr. Pinto)
- AWT Expert Committee "Residual Stresses", FA 13 (Dr. Pinto, Prof. Pyzalla)
- DGM working group "Tomography" (Prof. Borbely, Prof. Pyzalla)
- COST action 536 (Dr. Garcia, Dr. Weber)
- Thermo-Calc (Dr. Garcia, Dr. Weber)

In addition members of the department served the scientific community within a number of committees:

- member of the senate of the DFG, elected summer 2008 (Prof. Pyzalla)

- member of the supervisory board of TU Wien, elected December 2007 (Prof. Pyzalla)
- member of the "wissenschaftliche Kommission Niedersachsen", since 2007 (Prof. Pyzalla)
- member of the DFG „Fachkommission“ and „Bewilligungsausschuss“ for the German Excellence Initiative „Exzellenzinitiative“, since 2005 (Prof. Pyzalla)
- member of the panel "Products and Process Engineering" of the ERC for selecting the ERC Advanced Grants, 2008 – 2012 (Prof. Pyzalla)
- deputy chairperson of the "Photon Science Committee" (PSC) of DESY, since 2004, 2<sup>nd</sup> period started in 2008 (Prof. Pyzalla)
- member of the scientific advisory board of IPP, Garching, Germany, since 2008 (Prof. Pyzalla)
- member of the scientific advisory board of GKSS Geesthacht, 2005 – 2007 (Prof. Pyzalla)
- associate Member of the committee for neutron research (KFN) 2005-2007 (Prof. Pyzalla)
- member of the Kuratorium of "Welt der Physik", since 2008 (Prof. Pyzalla)
- member of the scientific advisory board of the DGM, since 2005 (Prof. Pyzalla)
- member of the "Preiskomitee II" of DGM, since 2007 (Prof. Pyzalla)
- member of the scientific committee of the German research neutron source FRM II (Dr. Pinto)

## Education of Ph.D. Students

### Ph.D. theses submitted

The Ph.D. students in the department "Material Diagnostics and Steel Technology" have submitted or will submit the following 5 theses to Ruhr-Universität Bochum, Faculty of Mechanical Engineering in 2008:

- Leonardo Agudo: "Influence of Alloying Elements on the Microstructure and Mechanical Properties of Steel-Aluminium-Joints Produced by Metal Arc Joining with Special Focus on the Intermetallic Phase Seam" submitted in June 2008, oral exam is due in November 2008.
- Rodrigo Coelho: "Joining of Light-Weight Materials by Friction Stir Welding and Laser Beam Welding", submitted in June 2008, oral exam is scheduled for November 2008.
- Augusta Isaac: "In-situ Tomographic Investigation of Creep Cavity Evolution in Brass" will be submitted in October 2008.
- Claudia Juricic: "Mechanisms of Internal Stress Formation in Iron Oxide Scales", will be submitted in October 2008

- Pedro Silva: "Microstructural Investigations of Fe-base Abrasion Resistant Coatings Co-Extruded on Steel Substrates", will be submitted until the end of the year 2008.

### Training for Ph.D. students

#### *Seminars*

Once every six weeks each Ph.D. student of the department "Material Diagnostics and Steel Technology" gave a report on the current state of his/her Ph.D. project to all members of the department. Additionally lectures were scheduled before oral presentations at national and international conferences.

#### *Thermo-Calc and Dictra courses*

Modern alloy development often necessitates the use of computational tools. Computational Thermodynamics are a powerful tool for alloy development, especially in the field of iron-based materials. The department "Material Diagnostics



and Steel Technology” was requested to continue the activities of Prof. G. Inden in this field and thus, the group for “Engineering Thermodynamics” was established, providing the service for Thermo-Calc and Dictra for the institute and working closely together with the group “Metallurgy”. Initiated by Dr. Garcia, internal Thermo-Calc and Dictra teaching courses were organized for the Ph.D.-students of the department. A first course on Thermo-Calc was given in October 2007, additional lectures were held by Prof. Inden. Based on the knowledge of the Thermo-Calc courses, in March 2008 courses for Dictra were started, again with the support of Prof. Inden.

All courses were intended to provide basic knowledge on thermodynamics and kinetics as well as the necessary skills to work with the aforementioned programs. The Ph.D.-students took advantage of these fresh skills to use them immediately for their own research projects, e.g. the development of new concepts for creep resistant steels or the processing of high-Mn steels.

*Participation of Ph.D. students in workshops, national and international conferences*

Ph.D. students were strongly encouraged to present their results in oral talks and posters at national and international conferences. Care was taken to allow each Ph.D. student at least once a year to attend a conference and to give those Ph.D. students in their 3<sup>rd</sup> year the opportunity to give oral talks at international conferences in order to further

improve their chances to obtain Post-Doc positions in scientifically well recognized groups.

*Participation of Ph.D. students and technical staff in training courses*

Ph.D. students and technical staff were encouraged to participate in external training courses. These included for instance

- Course on high resolution transmission electron microscopy (EMAT Winter Workshop, Training on TEM spectroscopy) at Universiteit Antwerpen, January 2008, Dr. Kostka and Mr. Agudo,
- Small Scale Plasticity Summer School, Braunwald, Switzerland, September 2007, Dr. A. Kostka
- VI-PNAM autumn school on “Application of Neutrons and Synchrotron Radiation in Engineering Materials Science”, autumn 2006, Augusta Isaac, Federico Sket, Marcin Moscicki and Krzysztof Dzieciol (2007)
- IDL Software course in Munich, March 2007, Augusta Isaac, Krzysztof Dzieciol, Federico Sket,
- Cost 536 Training School in Lanzarote, October 2008, David Rojas

*Further education of technical personnel*

- Course on Matlab GraphicUserInterface I and II, Aachen, 2007, Benjamin Breitbach
- Examination Radiation Protection Officer, Essen, 2007, Benjamin Breitbach, Dr. Haroldo Pinto
- English courses in Metallurgy (Ph.D. students)

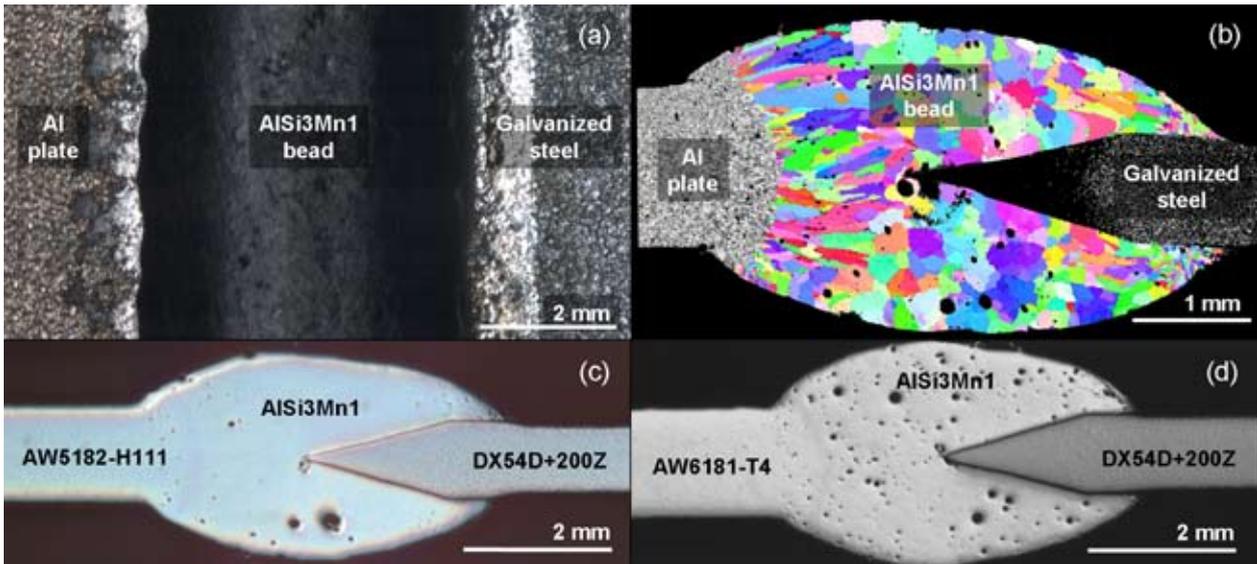
## Research Topics

### Metallurgy (S. Weber)

Within the department of “Material Diagnostics and Steels Technology” the group “Metallurgy” focuses on steel development with scientific as well as industrial background. Besides new alloys for flat products, also cast steels, e.g. tool steels, are investigated and developed based on a sophisticated analysis of microstructure-property relations.

Powder metallurgy is used as a complementary method to conventional metallurgy for special problems. The challenge is to find the best suited method for a specific application and to correlate differences between conventionally and powder metallurgically produced materials with microstructural features. All developments are based on computational thermodynamics using Thermo-Calc and Dictra in close cooperation with the workgroup “Engineering Thermodynamics” of Dr. José Garcia. The group started its work in February 2007 with the employment of Dr. Sebastian Weber as its head.

The strategy of the group “Metallurgy” is focussed on two different aspects. First of all, the needs of the steel companies organized within the Steel Institute VDEh are to be covered by solving problems and challenges with technical and industrial background. Therefore, the direct contact to the study groups (Arbeitsausschüsse) of the Steel Institute VDEh was reactivated and intensified. Additionally, the group started to participate in several co-financed research projects together with several industrial partners. The second focus affects the scientific work of the group on light elements in steels, particularly nitrogen, hydrogen and boron. Several activities were started on hydrogen in austenitic steels and also new concepts for a use of nitrogen as alloying element in steels are under investigation. Together with the group of Dr. Garcia, research activities on the influence of boron and nitrogen on the creep resistance of ferritic-martensitic steels were started. All of these activities are considering technical and technological aspects. The ambition is to establish a scientific group with international reputation in the field of light elements and interstitials in iron-base alloys.



**Fig. 1:** Macrographs of steel to aluminium alloy CMT joints: (a) Upper view of the joint; (b) Cross section image quality map (EBSD) of a CMT butt joint; (c) Cross section view of CMT DX54D+Z200/AW5182-H111 joint; (d) Cross section view of CMT DX54D+Z200/AW6181-T4 joint.

### Key Projects

#### Welding of steel to aluminum by cold metal transfer

The need for lightweight constructions in automotive industry has produced a strong interest in multi-material systems. Thus, joining of dissimilar materials, for instance steels to aluminium alloys, has become a necessity. Today most steel/Al-joints suffer from difficulties in substance-to-substance bonding arising mainly from the differences in the physical and mechanical properties of the parent materials and from the formation of brittle intermetallic aluminium-rich  $Fe_xAl_y$ -phases.

The disadvantages of conventional methods for joining steel to aluminum may be overcome using the so-called 'Cold Metal Transfer' (CMT) process, which is a modified metal inert gas welding technique for steel to aluminum welds and was introduced by Fronius International GmbH, Wels, Austria. In the CMT technique the work pieces to be joined remain considerably 'colder' than in conventional 'Gas Metal Arc' (GMA) processes. Due to the recent appearance of the CMT technology in the market, there is little knowledge about weld microstructure, the fundamental interfacial interactions between the parent materials and how the process parameters affect them.

Within this work in-depth investigations of the microstructures of different steel to aluminium joints were performed. The work was focused on relating the processing parameters of CMT with the resulting microstructures by applying for instance EBSD (Fig. 1). The formation of intermetallic phases in the weld seam was analysed by EBSD as well as TEM for different parent and filler materials in different

welding geometries. The results were related to mechanical properties of the joints. Amongst others it was shown, that the hindering effect of Si on the growth of the phase  $\eta-Al_5Fe_2$  is already very strong at early growth stages.

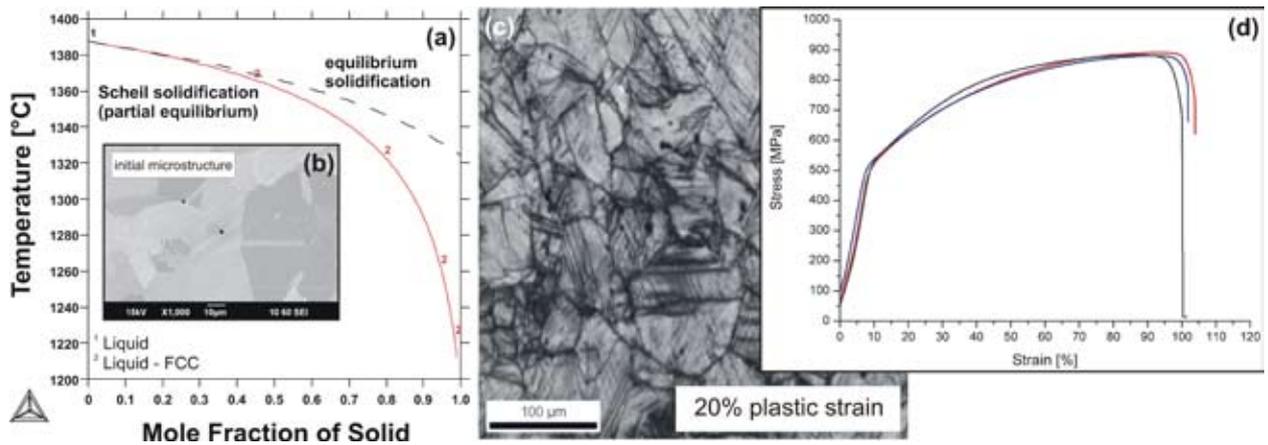
The CMT welding process has shown to produce aluminium-to-steel hybrid joints in both the single lap and butt configuration, the later case of which has been proven a reliable quality from the mechanical point of view. Thus, the CMT process represents a viable alternative when considering the implementation of fusion welding techniques for the production of hybrid pieces in the automotive industry [1-4].

#### Development of corrosion resistant TWIP-steels

In recent years several new austenitic steels within the system Fe-Mn-Al-Si-C were developed and characterized concerning their mechanical properties. Depending on the alloy composition and the temperature a change of the predominant deformation mechanism, including TRIP (transformation induced plasticity) and TWIP (twinning induced plasticity), is possible. Due to their mechanical properties these steels are very interesting for crash resistant parts.

On the other hand, within the group of austenitic stainless steels new alloys were developed emerging from the system Fe-Mn-Cr-Mo-C-N. In this context the positive influence of a combined alloying with C and N is well known. Using computational thermodynamics it was possible to develop high nitrogen steels bearing the necessity of pressurized metallurgy.

The scope of the research project is to develop a corrosion resistant steel exhibiting twinning induced



**Fig. 2:** Corrosion resistant TWIP steel: (a) Results of an equilibrium and a Scheil calculation with partial equilibrium for C and N resulting in a primary austenitic solidification; (b) Initial microstructure exhibiting a coarse grain size and annealing twins; (c) Microstructure after 20% of plastic strain showing a high density of deformation twins; (d) Engineering stress-strain curves.

plasticity (TWIP) within the system Fe-Mn-C-N-X-Y whereas the components “X” & “Y” are used to tune the nitrogen solubility of the melt, control the solidification path and influence the stacking fault energy. The works started with thermodynamic calculations using Thermo-Calc, followed by casting experiments to gain appropriate materials (Fig. 2). The new materials were characterized concerning their microstructure and mechanical properties, followed by the development of an appropriate heat treatment. Current work is focusing on the simulation of precipitation kinetics with Dictra and its experimental validation.

### Engineering Thermodynamics (J. Garcia)

The mission of the group Engineering Thermodynamics is the development of modern steels and iron-based alloys for engineering applications assisted by thermodynamic computational methods. The information provided by the thermodynamic and kinetic calculations is employed to reduce the trial-and-error process in the development of novel Fe-alloys. For this purpose the group works in three main fields: 1) basic research and assessments, 2) development of master and multi-component alloys (thermodynamic calculation and production of prototypes) and 3) modelling of processes (in laboratory and industrial up-scaling). Regarding the first area, efforts are being carried out in the determination of basic phase diagrams and equilibrium phases coupling experimental investigations and thermodynamic calculations. Ongoing activities on the re-assessment of technological important phase diagrams i.e. Fe-Mn-W-C, Fe-Mn-W-Si-C or Co-W-Ti-C for the production of steels and steel-based alloys must be remarked. For the development of master alloys, the influence of alloying elements on i.e. the austenization temperature, or the phase formation, as well as the heat treatment conditions, is currently modeled. The equilibrium calculations

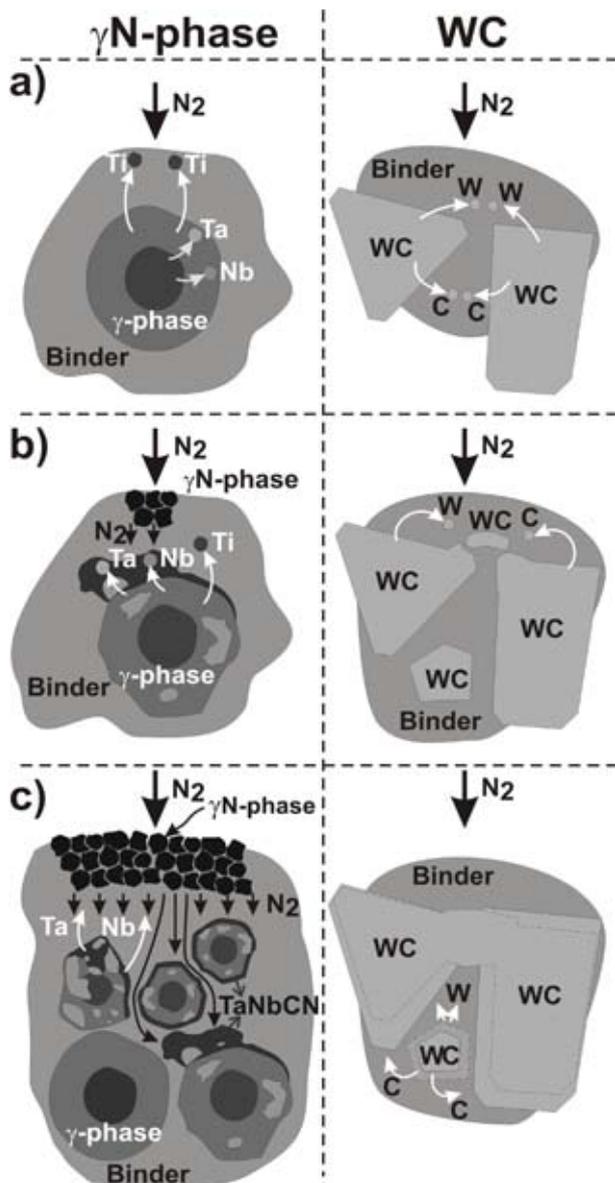
are coupled with another key activity of the group: the calculation of kinetically-controlled processes for phase transformation, phase precipitation and coarsening or grain growth. These calculations are widely employed for the ongoing development of novel 9-12%Cr creep resistant steels. Other processes including interactions of metal/metal, metal/carbide and metal/gas for thermal treatments and diffusion-controlled thermal surface treatments are also relevant. Nitridation of novel Fe-based composites are modeled and within this information successfully industrial scaling-up processes were carried out. The group employs the commercial software Thermo-Calc and Dictra with up-dated databases i.e. TCFE5, SSOL4, SSUB3, etc. The experimental techniques are assisted and supported by the metallurgy group of Dr. Sebastian Weber and his co-workers in very close cooperation.

The group Engineering Thermodynamics is the official contact with the Swedish company Thermo-Calc AB for the installation and update of the software and provides the Thermo-Calc and Dictra service for the entire institute. The group is totally integrated to the CALPHAD organization and has an active participation on this community. The group also has a strong cooperation with the ACCESS group (German agent for Thermo-Calc AB), which is associated with the RWTH in Aachen, and is a vigorous participant of the activities carried out by this institution.

### Key Projects

*Design of novel 9%Cr heat resistant steels assisted by thermodynamic calculations*

This project aims at developing 9%Cr heat resistant alloys for temperature service conditions above 650°C in power plants (key factor for reduction of CO<sub>2</sub> emission). The concept is to find appropriate additions of Boron and Nitrogen in order to obtain



**Fig. 3:** Schematic modelling explaining the preferred dissolution process of Ti, Ta and Nb MX carbonitrides for the formation of graded surfaces (in the presence of a high Nitrogen activity) during heat treatment of Fe-based composites [10].

fine precipitation of MX carbonitrides, control of Laves phase formation, and to avoid both the coarsening of i.e.  $M_{23}C_6$  and the formation of detrimental Z-phase. The influence of the addition of alloying elements is modeled by thermodynamic calculations. Upon these calculations, master alloys are produced in cooperation with the metallurgy group. Tension, compression and creep tests at varying loads and temperatures are carried out. The alloys are fully characterized by high resolution microscopy techniques not only after the production step but also at the different creep stages [5].

*Modelling of kinetics of phase transformation in heat resistant steels*

In this work the kinetics of phase transformation in multi-component systems are modeled by DICTRA. This work is based on previous activities on field of modelling of phase formation and phase equilibrium on heat resistant steels by Prof. G. Inden and Dr. A. Schneider, which consider the competition between stable and metastable phases. Long term creep conditions can be modeled including, i.e., coarsening of carbonitrides, growth of Laves phases and eventually the formation of Z-phase. The results of the modelling are compared with experimental data. Ongoing calculations are being carried out for the systems Fe-Cr-W-C-N-B and novel ferritic-martensitic heat resistant steels.

*Alloy development for critical components for power plants*

Within this project [6], master alloys of basis Fe-Cr-X are produced in order to investigate the influence of Boron in the phase formation and phase stability of creep resistant alloys [7]. Also the influence of non-conventional elements such as La, Ce, Nd but also Ta and Hf is investigated. The production of the master alloys is supported by the new laboratory "Spark spectroscopy for materials processing" in cooperation with the group "Metallurgy". The development is assessed by thermodynamic and kinetic modelling.

*Development of nano-scaled Fe-alloys and composites*

The processing of iron and iron-base bulk alloys with nano-scale grain size (around 20 nm) is investigated [8]. A new process, combining a wet chemical sol-gel route (based on precursors of tri-block copolymers and iron alkoxides organic solutions), with an additional consolidation step is applied. Thermodynamic modelling on the field of crystal defects and grain boundaries in cooperation with the group of Prof. Dr. Armando Fernandez-Guillermet in CAB-Argentina is foreseen. By means of TEM, phase formation, particle size, particle distribution and coarsening will be investigated. Mechanical (E-module, hardness) and thermo-physical properties (heat capacity, Curie temperature) of the nano-Fe materials are compared with corresponding Fe-materials of conventional grain size.

*Thermodynamic modelling of the interaction of Fe-X alloys with MeC*

The interaction between Fe-X-based alloys with carbides of the type MeC (with Me: W and/or Ti)



are investigated. Phase equilibrium thermodynamic calculations of the systems Fe-C-X-Me (as a function of C content) are carried out. The objective is to avoid the formation of phases, which are detrimental for the overall mechanical properties such as  $M_6C$  carbides and graphite. Modelling of inter-diffusion mechanisms in the steel-carbide systems is carried out with Dictra considering a multi-component model. The basic research is to be applied for the development of reinforced Fe-Mn alloys or Fe-X-Me-C composites.

#### *Modelling of formation of graded outer-surfaces on composites with Fe-based matrices*

The formation of diffusion-controlled wear and corrosion resistant graded layers in W-Ti-Ta-Nb-C cemented carbides with Fe-based matrices is investigated [9]. Nitridation results in the formation of a very fine-grained (Ti,Ta,Nb)(C,N) phase with inclusions of binder and WC in the near-surface of the cemented carbides. The high nitrogen activity at the surface during the nitridation treatment is the driving force for the formation of the (Ti,Ta,Nb)(C,N) outer layer. The selective diffusion of Ti, Ta and Nb in the presence of high N activity results in the preferred dissolution mechanism for the core-rim structure, leading to the formation of gradients of Ti and Ta within the (Ti,Ta,Nb)(C,N) outer phase. A kinetic/phenomenological model for the formation of the graded layers is proposed. Ongoing Dictra calculations considering different binder matrices are being carried out [10]. The results are of technological relevance for the adjustment of the overall compositions and processing steps.

#### **Microstructure Characterization (A. Kostka)**

Demands on economical and ecological aspects in automotive and aerospace industry generate the development of advanced engineering materials and continuously increase in light-weight design of manufactured parts. Moreover, the industrial request to weld lightweight materials such as aluminium alloys to steels requires application of new welding technologies, which very often suffer from lack of microstructural information.

Metallurgical reactions during heating and cooling as well as plastic deformation are mainly responsible for the microstructure and properties of metallic engineering materials. These reactions have been studied in great detail for many decades to establish optimum chemistry and thermomechanical processing treatments for a wide variety of materials. However, when engineering metallic materials are joined, the localised weld thermal and hot deformation processes alter the optimized base metal properties. The result is the creation of non-equilibrium microstructures in

the joint region, which are significantly different in both morphology and properties from those found in the base material.

Recently new joining technologies (like friction stir welding or explosion cladding) offer metallurgical bonding of dissimilar materials with strength which is satisfactory from the engineering point of view, but at the same time the basic mechanisms occurring at the interface are not fully understood or even remain unknown. Therefore the scientific interest of the group is focused on systematic investigations and understanding of the metal-physics phenomena occurring at the interface of the dissimilar joints, formation and evolution of microstructures of advanced engineering alloys during processing, creep and joining. This knowledge is becoming increasingly important as the foundation for the microstructure-based modelling of materials and component behaviour under complex states of stress, strain and temperature.

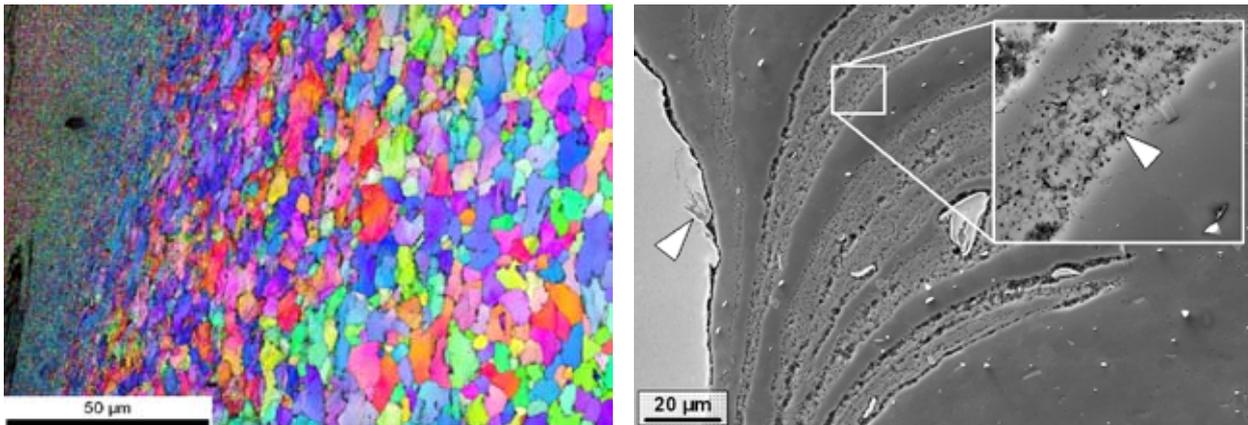
The complexity of metallurgical phenomena covers a broad spectrum of knowledge which create the opportunities for a number of future projects aiming at the extending of the generated knowledge and/or addressing new challenges in joined structures. There is a need to enhance the available knowledge on the microstructural features occurring during materials processing and interfacial reactions occurring in dissimilar joints and to integrate this understanding with the production processes.

The focus of the group “Microstructure Characterization” in the department “Material Diagnostics and Steel Technology” is on microstructure analyses and correlations between microstructure and (mainly mechanical) properties of steels and dissimilar welds. An example of the microstructures formed by friction stir welding is presented in Fig. 4.

Currently dissimilar welds between steels and light metals are of particular interest for the group. The aim of the research performed is to understand the metal – physics phenomena of the interface of the dissimilar welds and to determine the deformation mechanisms and their spatial inhomogeneities occurring during the deformation of dissimilar welds. Based on these experiments the group aims at contributing to an optimization of dissimilar weld.

The Microstructure Characterization group provides wide service of experimental investigations using scanning and transmission electron microscopy (including a specimen preparation for Transmission Electron Microscopy) for the others groups of the department and Institute.

At the moment, the microstructure of welds of dissimilar configurations is in focus, but in the future



**Fig. 4:** Left hand side: Orientation map (taken by EBSD) of the friction stir welded AA6181-T4 aluminium alloy to HC340LA high strength steel (single overlap configuration) showing a gradient of the grain size from the steel base material, heat affected zone and thermo-mechanically affected zone resulting from accumulated strain. Right hand side: SEM image showing an interface of FSW joint produced in but-joint configuration. Arrows indicate formation of different intermetallic phases:  $Fe_xAl_y$ -compound formed at the aluminium alloy - steel interface and  $Al_xZn_y$ -compound present in the AA6181-T4 aluminium alloy as a result of the Zn incorporation from the steel plate surface.

more attention will be put to the aspects associated with the plastic deformation of materials like studies on the dislocations, their nucleation, mobility and interactions as very attractive for research on plasticity.

#### Key Projects

##### *Formation of the intermetallic $Fe_xAl_y$ phases in a steel Al-alloy welds*

This project aims at comprehending the formation mechanisms and growth mechanisms of the aluminium rich intermetallic  $Fe_xAl_y$ -phases at the interface of diffusion couples, immersion tests and steel/aluminium welds and the correlation of the interfacial reaction products with the joining process parameters.

Moreover, an extension of this project is provided as a scientific cooperation with BIAS (Bremer Institut für angewandte Strahltechnik GmbH), where the goal of the research is the development of a physically reasonable model that represents the growth of the intermetallic phase layer as a result of thermal welding, which is to be integrated in an FEM based thermal-mechanical simulation of the welding process. The MPIE role in this project will be associated with the characterization of the microstructure and the understanding of its evolution during welding at different conditions.

##### *Metallurgical phenomena in friction stir welded joints*

The main strategic research topic of the Virtual Institute (VI) IPSUS (Improving Performance and Productivity of Integral Structures through Fundamental Understanding of Metallurgical Reactions in Metallic Joints) is the understanding of nonequilibrium metallurgical phenomena (i.e. precipitation, recrystall-

sation and phase transformation under high heating and high cooling rates) in metallic joints on light-weight materials, using in-situ synchrotron-based diffraction techniques. The VI-IPSUS will generate a unique knowledge, especially as far as the kinetics of recrystallisation and precipitation in welded joints in Al and Mg alloys is concerned. In the case of in-situ monitoring of phase transformations the uniqueness of the VI-IPSUS lies on:

- Understanding of complex metallurgical phenomena inherent to the advanced steels to be investigated, i.e. twinning induced plasticity (TWIP) steels and oxide-dispersion-strengthened (ODS) steels;
- Phase formation in dissimilar joining of these steels with light-weight materials, especially to Al alloys.

##### *Physical-metallurgical reactions governing explosion cladding*

This project focuses on the understanding of the bonding mechanisms occurring during the explosion cladding of dissimilar materials. Major emphasis is placed on microstructural investigations of the physical-metallurgical reactions like phase formation, defect configuration and densities and the correlation of the microstructure with the mechanical properties of the joints.

#### Materials Testing (H. Pinto)

The main focus of the activities within the research group Materials Testing is to develop non-destructive methods capable of characterizing complex multiphase microstructures regarding their chemical and structural gradients, the anisotropy of their elastic and plastic deformation behaviour and the formation and distribution of their internal stresses. The strategy is focused on the further development of non-destructive diffraction techniques with the



sensors X-rays, synchrotron X-rays and neutrons. The link to the macroscopic mechanical performance of engineering materials is provided by traditional mechanical testing.

Laboratory X-ray sources enable microstructure and stress analyses within the near-surface zone. The use of grazing incidence techniques allows furthermore the assessment of gradients of phase composition and internal stresses in thin films. Synchrotron radiation and neutrons are chosen for the characterization of material properties in the bulk. Whereas high-energy synchrotron radiation is convenient for the study of light materials and simple sample geometries, neutrons allow the analysis of technical components with more complex geometries and samples containing heavier elements. New perspectives concerning in-situ studies of physical and mechanical material properties as well as microstructural damage due to external loads result from the high photon flux, the high collimation and the broad energy spectrum of the synchrotron radiation. Thus, high spatial, depth and time resolution become achievable for studying the time evolution of complex microstructures and their properties.

The group is equipped with a multi-task X-ray lab for phase, stress and texture studies, several mechanical testing machines such as Universal mechanical testing, creep testing and microhardness as well as with furnaces for heat-treatments. A resistance furnace for oxidation in air at temperatures up to 1100°C as well as a miniaturized tensile testing device for uniaxial straining was developed by the group for in-situ studies on the diffractometers at MPIE as well as on diverse synchrotron XRD setups at e.g. the Materials Science beamline EDDI of HZB at BESSY, the high-energy materials science beamline HARWI-II of GKSS at HASYLAB, the Material X-ray IMaging (MAXIM) beamline G3 of the HASYLAB or at diffraction instruments of the ESRF (ID31, ID15 and BM20). The Materials Testing group provides furthermore service in terms of XRD measurements and mechanical testing for the other research groups at the institute.

### Key Projects

#### *Texture, strain and stress development in oxide scales [11-14]*

The integrity and adherence to substrate of protective oxide scales are impaired by stress generation accompanying the growth of oxides on metallic surfaces. Several mechanisms of stress formation during oxide growth have been proposed over the past years. However, none of those processes can solely explain the internal stress situation in most of the naturally growing oxide scales. This project funded by the FWF, Austria, and the MPIE focuses on the particular influence of substrate orientation and

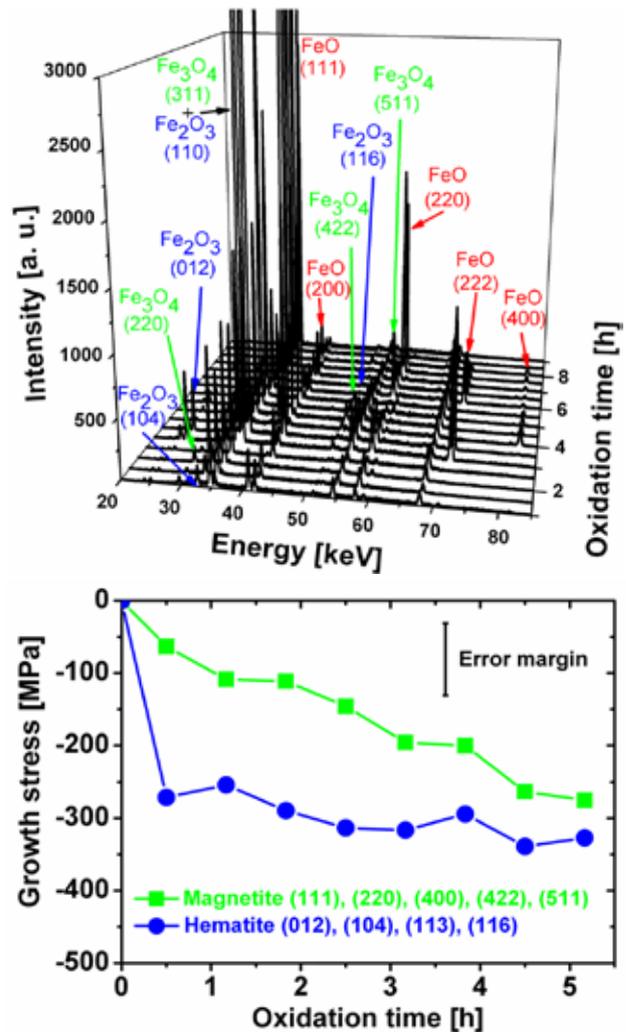


Fig. 5: top: Typical energy-dispersive diffractograms obtained during in-situ oxidation of iron substrates at 650°C, beamline EDDI at BESSY; bottom: time evolution of phase-specific growth stresses during iron oxidation at 450°C.

preferential orientation of oxide growth on the internal stress state of multiphase oxide scales. To this end, the time evolution of microstructure, internal stresses and their gradients in oxide scales growing on polycrystals and single crystals of pure iron and Fe-Al alloys are studied in-situ during oxidation by energy-dispersive XRD with synchrotron radiation (Fig. 5) and by complementary electron microscopy.

The results reveal an increasing influence of substrate orientation on the internal stresses of oxide scales with increasing oxidation resistance, i.e. decreasing scale thicknesses. Oxide scales growing on Fe-Al alloys at a low temperature (700°C) evolve therefore highly anisotropic internal stresses due to epitaxial growth. Also the volume fraction of metastable less-protective alumina phases strongly depends on the type of substrate. The internal stress state in iron oxide scales, on the other hand, is governed by concurrent processes of inner oxide formation, inhomogeneous pore formation as well as creep of the oxides and the substrate.

*Induction assisted welding technologies in steel utilisation [15-16]*

HSLA steel grades are required to reduce weight in industrial machinery. Their mechanical performance is impaired by welding operations which often cause a reduction of toughness and increase the probability for cold cracking due to martensite formation in the fusion zone (FZ) and heat-affected zone (HAZ). Induction heating is capable of directly producing heat inside a work piece. This enables the integration of induction heat-treatments into serial welding processes.

In this project, the effect of induction-assisted laser welding on the microstructure and residual stresses of HSLA steel joints was investigated within the framework of the EU-project INDUCWELD funded by the 'Research Fund for Coal and Steel'. The results reveal that conventional laser welding causes strong martensite formation in the FZ and HAZ. This leads to prohibitive hardness values. Integrated induction heat-treatments resulted in an efficient hardness reduction in both regions. The residual stresses after laser welding with induction heating are typical for fusion welding and still of significance. The use of adequate fillers to lower the transformation temperatures is a possibility to reduce the residual stresses in the FZ and HAZ.

*Defects and internal stresses in novel austenitic high-Mn steels*

The deformation behaviour (TRIP/TWIP) of austenitic high-Mn steels is influenced by the steel composition and the temperature which change the stacking fault energy (SFE) of the austenitic lattice. In addition to the SFE, the level of deformation and the multiaxiality of the stress state also affect the deformation mechanisms of the austenite. On the other hand, the manufacturing and service of mechanical components from high-Mn steels are accompanied by the formation of macro- as well as micro stresses. The positive and negative effects of macro stresses on the performance of mechanical components are well-known. The impact of micro stresses on damage initiation remains, however, fully unclear. High micro stresses are expected to occur in high-Mn steels due to the thermal expansion mismatch and the differences in mechanical behaviour of the phase constituents as well as due to strain-induced phase transformation. Furthermore, the elastic and plastic anisotropy of the austenite associated with texture formation cause inter- and intragranular stresses during forming.

This project is part of the collaborative research unit SFB 761 – Steel ab-initio – and focuses therefore on understanding the connection between the evolution of phase composition, defect configurations and

internal stresses, and the mechanical behaviour of high-Mn steels during uni- and multiaxial loadings. The work started with the study of the mechanisms of micro residual stress formation in TRIP alloys during uniaxial straining.

*Wear protecting nanostructured coatings for cutting processes*

The productivity as well as the life-time of cutting tools can be considerably increased by combining suitable technologies to modify the surface of the substrate prior to coating coupled with new multiphase nanostructured coatings.

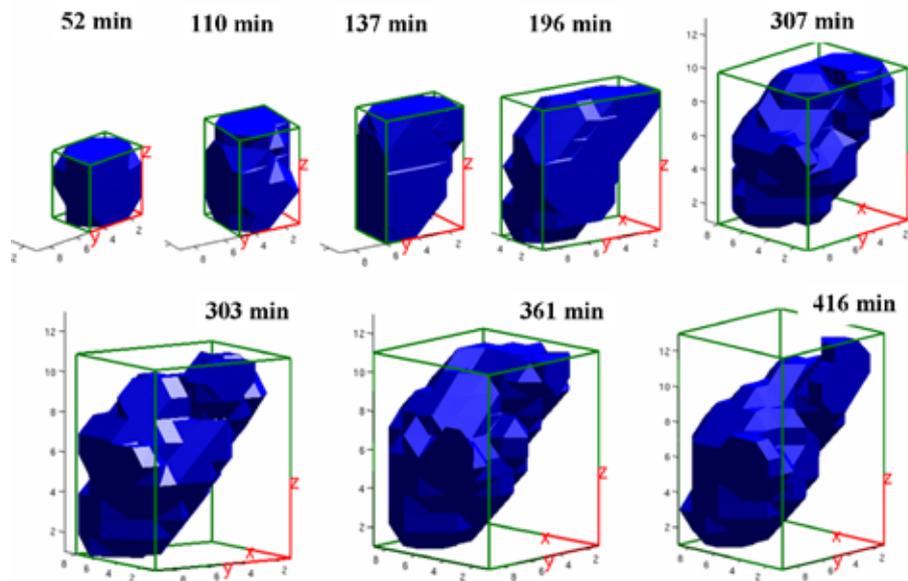
This project is a DFG research grant for German-Brazilian cooperation aiming at understanding the mechanisms of wear, corrosion, phase transformations and damage initiation in novel wear protecting nanocoatings. Dr. J. Garcia will guide the production of coatings with tailored microstructures (graded, multilayer and composite) by thermodynamic simulations and cutting tests. Dr. H. Pinto will be responsible for the microstructure and residual stress assessment in the coated tools. The Brazilian partners will pre-treat the substrates by noble gas bombardment (UNICAMP) and characterize the resulting microstructures and residual stresses of the modified substrates (Brazilian Synchrotron Light Laboratory – LNLS). The work started with ZrC-coatings deposited on 100Cr6 steel substrates.

**Synchrotron and Neutron Methods (A. Borbély)**

Experimental research of the Synchrotron and Neutron Methods group is related to recent developments at synchrotron sources, which represent a guarantee for obtaining new and relevant information. The research is focused on the characterization of the real three-dimensional structure of metals undergoing plastic deformation and can be divided in two main parts:

- a) Application of in-situ microtomography to damage characterization during high-temperature creep of metals, and
- b) Further development of the three-dimensional X-ray diffraction (3D-XRD) method for evaluation of internal stress in single grains of polycrystalline materials.

Our strategy concentrates on time resolved investigations allowing an insight into the dynamics of the studied processes. Most of the results were obtained from collaborations with beamlines ID15 and ID19 at the European Synchrotron Facility (ESRF) in Grenoble, and beamlines HARWI-II and F1 of the Hamburger Synchrotronstrahlungslabor (HASYLAB) at Deutsches Elektronen-Synchrotron (DESY).



**Fig. 6:** Typical example of cavity shape evolution as a function of creep time. The last three stages belong to accelerated creep.

### Key Projects

#### *In-situ tomographic investigation of creep*

Combining microtomography with image analysis we have shown that the tomographic method is adequate to characterize local changes of the sample's geometry during creep, making possible the evaluation of local creep-curves for tiny material slabs with heights of about  $10\ \mu\text{m}$  [17]. This local kinematical information could be exploited to evaluate the activation energy of steady-state creep in brass [17]. By imposing a constant temperature gradient along the tensile axis of the sample, material slabs at different heights have different temperatures and consequently different strain rates. It was shown that the tomographic technique is extremely accurate, so that differences in local deformation among small material volumes can be detected. The activation energy of steady-state creep determined on a single small specimen (with volume of about  $1\ \text{mm}^3$  and constant temperature gradient) had a similar value as reported in the scientific literature, but obtained by testing many large specimens at different temperatures [18].

#### *Determination of the growth rate of single cavities*

Application of image correlation techniques to successively reconstructed volumes proved to be an excellent tool for monitoring damage development. The technique was used to follow the position and volume evolution of single cavities during creep of leaded brass. Other nondestructive techniques, like X-ray or neutron small-angle scattering, deliver only statistical averages of the voids ensemble and the evaluation of their spectra is rather influenced by assumptions made about the void shape. Microtomography in contrast delivers the true 3D shape

of the voids without any extra assumptions. Using software developed in our group we could evaluate the growth rate of more than 6 000 single cavities from the detection limit (diameter of about  $2\ \mu\text{m}$ ) until coalescence [19,20]. One typical example of cavity volume evolution is shown in Fig. 6. The analysis of this representative dataset proved that the classical "power-law creep" mechanism proposed by Cocks and Ashby [21] is able to describe of about 46% of the detected individual growth rates. Finite Element Modelling (FEM) of the creep deformation showed in addition that plasticity and the resultant higher local stress-triaxiality around voids can explain other 40% of the growth rates experimentally quantified. The remaining 13% was attributed to grain boundary-sliding, a mechanism which is enhanced in the studied case by the existence of a liquid lead phase wetting the grain boundaries. The results obtained have important theoretical significance being the first direct check of a cavity-growth mechanism.

#### *Indexing single grains with 3D-XRD*

Another novel method developed at synchrotron sources is the three-dimensional X-ray diffraction technique (3D-XRD). The method initially proposed by Poulsen [22] uses the penetrating power of high-energy beams and is capable to deliver crystallographic information from single grains embedded in the bulk of polycrystalline materials. 3D-XRD was further developed in our group in order to increase the accuracy of the evaluation of orientation and spatial position of the grains. A first test of our software was performed on diffractograms acquired during in-situ tensile straining of austenitic steel specimens. Plasticity induced orientation changes in single grains of an X10Cr-Ni18-8 steel sample showed similar characteristics as those in aluminium deformed under similar conditions [23].

## Outlook

Prof. Dr.-Ing. Anke Pyzalla has left the Institute on September 30th, 2008 to take an employment as scientific general manager of the Helmholtz-Zentrum Berlin für Materialien und Energie GmbH. The outlook for the individual research groups are:

1. The major activities (ongoing Ph.D.-works and research projects) of the former group Metallurgy are continued at the Ruhr-Universität Bochum, Institute for Materials, in Bochum. In September 2008 the Max-Planck-Institut für Eisenforschung and Dr. Weber agreed on a premature termination of his contract to give him the possibility to start in a new position at the HZB on 1<sup>st</sup> of October. This change was associated with the new position of Prof. Pyzalla at the HZB. The responsibility for the service activities of the group "Metallurgy" was transferred to Dr. Ponge from the Microstructure Physics and Metal Forming (MU) department at that time.
2. The activities (ongoing Ph.D.-works and research projects) of the former group Engineering Thermodynamics will be continued at the Ruhr-Universität Bochum. In September 2008 the Max-Planck-Institut für Eisenforschung and Dr. Garcia agreed on a premature termination of his contract to give him the possibility to start in a new position at the HZB on October 1<sup>st</sup>. This change was associated with the new position of Prof. Pyzalla at the HZB.
3. The group Microstructure Characterization will continue the ongoing projects regarding the characterization of dissimilar welds with focus on the interfacial reactions, growth and evolution of the intermetallic phases as well as on the experimental visualisation of the material flow during friction stir welding. Research of the microstructural aspects of plasticity of advanced engineering materials: mechanisms of deformations, nucleation, mobility and interactions of dislocations will be initiated in co-operation with the MU Department.
4. The group Materials Testing will carry on the methodological development of non-destructive diffraction techniques towards the formulation of evaluation procedures for stress analysis taking texture, micro stresses and planar faults into account as well as the analyses of near-surface stress states with chemical and structural gradients. These new methodologies shall contribute to a better understanding of the stress-generating mechanisms during the growth of multiphase oxide scales, the connection between micro stresses and damage initiation in novel TRIP/TWIP steels as well as the mechanisms controlling damage in novel wear protecting nanocoatings.
5. Microtomographic investigations have revealed that the growth rate of cavities is size dependent. To quantify the growth of voids with sizes below micrometer, investigations with the newly developed nano-tomography method [24] are planned. A complementary study of these small voids will be performed by small-angle scattering methods, too. The 3D-XRD method is now further refined for the evaluation of the strain and stress tensor prevailing in each single grain during plastic deformation. Such a result will be also a premiere in the field of polycrystalline plasticity for which it is known that the classical Sachs [25] and Taylor [26] assumptions can be considered only as lower and upper bounds, respectively.

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## Research Projects in Progress

### Metallurgy

*Agudo, Kostka, Pinto, Weber*: Welding of Steel to Aluminium by Cold Metal Transfer

*Frommeyer, Weber*: Joint project: Development of corrosion resistant and dilation optimized microchannel heat sinks for high power diode laser – NextGen-, selection and qualification of materials

*Mujica, Weber*: Development of corrosion resistant TWIP-steels

### Engineering Thermodynamics

*Maccio, Garcia*: Thermodynamic modelling of the interaction of Fe-X-alloys with MeC

*Prat, Garcia*: Modelling of formation of graded outer-surfaces on composites with Fe-based matrices

*Rojas, Prat, Garcia*: Modelling of kinetics of phase transformation in heat resistant steels

*Rojas, Prat, Weber, Garcia*: Alloy development for critical components for power plants

*Weber, Garcia*: Development of nano-scaled Fe-alloys and composites

### Microstructure Characterization

*Coelho, Kostka*: Metallurgical phenomena in Friction Stir Welded joints

*Kostka*: Physical-metallurgical reactions governing explosion cladding

*Springer, Kostka*: Formation of the intermetallic Fe<sub>x</sub>Al<sub>y</sub> phases in steel Al-alloy welds

### Materials Testing

*Barbatti, Sales, Pinto*: Defects and stresses in novel high-Mn steels

*Brito, Pinto*: Phase composition and internal stress development in the oxidation of Fe-Al alloys

*Corpas, Pinto*: Microstructure and residual stress formation in induction-assisted laser welding of HSLA steels

*Friák, Lyemperakis, Fabritius, Pinto*: Internal stresses in lobster exoskeletons

*Friedel (TKS, Germany), Pinto*: Micro residual stresses in X-IP steels

*Juricic, Pinto*: Texture, strain and stress development in iron oxide scales

*Medeiros, Correia, Garcia, Pinto*: Wear protecting nanostructured coatings for cutting processes

### Synchrotron and Neutron Methods

*Borbély*: Microstructural investigations of the kinetics of strain accumulation and damage development during creep of short fiber reinforced aluminum alloys

*Dumont, Borbély*: The hierarchical structure of sauro-podomorph bones as a key to exceptional body size: Insights from materials science

*Isaac, Sket, Dzieciol, Borbély*: In-situ tomographic investigation of creep

*Isaac, Sket, Dzieciol, Borbély*: Determination of the growth rate of single cavities

*Moscicki, Pinto, Borbély*: Indexing single grains with 3DRXD

Members of the groups „Materials Testing“, Dr. Pinto, and “Synchrotron and Neutron Methods”, Prof. Borbély, were also successful in the competition for beamtime at major synchrotron sources and the neutron source of GKSS, Geesthacht. This resulted in beamtime allocations of:

- 27 days at ESRF, Grenoble, France
- 121 days at HASYLAB, Hamburg, Germany
- 21 days for test experiments at BESSY, Berlin, Germany in co-operation with Prof. Ch. Genzel, HMI and TU Berlin
- 7 days at the Brazilian Synchrotron Light Laboratory (LNLS), Brazil
- 6 days at FRG-I, Geesthacht, Germany





# Department of Materials Technology

G. Frommeyer

## Scientific Concepts

The department accomplishes research on a variety of novel structural and functional materials with special emphasis on iron-based alloys. A quantitative description of the relationships between materials processing parameters, microstructural features and related properties is one of the main objectives in addition to materials development.

The processing, based on diverse techniques can be performed successfully because of the excellent facilities of the collaborating departments. A detailed structural characterization on different microscopic levels down to the atomic scale provides a basic understanding of the microstructure evolution and the resulting materials properties. The focal point of the steel research is centred on high-strength and supraductile light-weight TRIPLEX steels and on fine-grained superplastic ultra-high carbon steels. Besides the ordered alloys based on iron-, nickel- and titanium aluminides the high melting point Laves phases are of great scientific and technological interest. The aluminides with superlattice structures exhibit potential capacity for high-temperature applications.

Functional materials like iron silicon/aluminium alloys with excellent soft magnetic properties and high permeability or rapidly solidified amorphous

iron/cobalt -silicon-boron ribbons and thin wires exhibiting the Giant Magnetic Resonance (GMR) effect are under current investigations and are of great interest for the electric power and electronic industry.

Analysis and modelling of continuous casting and rapid solidification process like in-rotating-liquid spinning (INROLISP), planar flow casting meltspinning (PFC), strip casting (SC) and laser welding have been performed in order to optimize the governing process parameter and to achieve a basic understanding for a fundamental quantitative description of continuous casting process operations. The table below shows the research topics of the groups in a two years time period.

The **Innovative Steel Research** group is headed by R. Rablbauer and provides the development and characterizations of microstructures and properties of new classes of high strength and supraductile steels possessing specific deformation and strengthening mechanisms based on metal physics principles. The research work is carried out in a fruitful cooperation with partners of the national and European steel industry. In the past two years the research work was mainly focused on high manganese aluminium carbon TRIPLEX steels with markedly reduced densities and excellent deep drawing and stretch

Research group	Scientific Topics in 2007 and 2008
Innovative Steel Research	Development and Characterization of high strength and supraductile light-weight steels, Super TRIP and TRIPLEX steels, and of low cost stainless steels exhibiting TRIP, TWIP and dislocation glide mechanisms
Rapid Solidification Technology	Nanocrystalline and amorphous iron- and cobalt-based materials with specific softmagnetic properties; Continuous casting of stainless steel wires
Ordered Alloys for High-Temperature Applications	Characterization of the defect structure and properties of DO <sub>3</sub> -, B2 and L1 <sub>0</sub> - ordered aluminides like Fe <sub>3</sub> Al-, NiAl- and γ-TiAl- based alloys and of TM Laves phases
Nanosopic Characterization of New Materials	Structural characterization of high manganese steels and ordered alloys with superlattice structures on the atomic scale by analytical TEM and atom probe field ion microscopy (APFIM)

forming properties, Super TRIP steel of ultrahigh strength and superior ductility, and low cost stainless steels exhibiting improved strength and plasticity due to complementary TRIP, TWIP and dislocation interactions. In addition ultrahigh strength pearlitic steels reaching almost the theoretical strength of iron have been developed for advanced tire cord applications.

The research group **Rapid Solidification Technology**, headed by J. Gnauk until August 2007, is working in close cooperation with the steel technology group. The main tasks are modelling of solidification and microstructural evaluation of near net shape casting processes. Also nanocrystalline and amorphous wires and ribbons of functional materials such as quasicrystalline Fe-Si-B or Co-Si-B alloys of high strength and specific soft magnetic properties with pronounced magnetoelastic anisotropy for the fabrication of highly sensitive measuring devices like transformer cores and sensors based on the Magneto-Electric Proximity (MEP) or Giant-Magneto-Impedance (GMI) effects have been produced by rapid quenching and characterized by strain induced magnetization. Another research activity is the development and process analysis of continuous casting of wires on by using modified twin roller facility. This constructional lay-out enables casting of several parallel aligned wire strands in one trail.

The aim of the research group **Ordered Alloys for High-Temperature Applications**, directed

by G. Frommeyer, M. Palm and F. Stein, is the development of creep and hot gas corrosion resistant refractory alloys based on intermetallic compounds with superlattice structures and their alloy constitutions represented by complex ternary or quaternary phase diagrams. The most promising high-temperature and oxidation resistant materials for structural applications in energy conversion facilities with improved thermal efficiency, such as steam or gas turbines and internal combustion engines are the aluminides of the iron group metals like  $Fe_3Al$  (Cr,Nb,Ti,W),  $FeAl$  (Cr,Zr,Mo) and  $NiAl$  (Cr,Mo,W) possessing the  $D0_3$ - or B2- ordered lattice structures, low density, high elastic stiffness. The greatest challenge in this field is the increase in room temperature ductility whereas the high warm strength and creep resistance can be preserved.

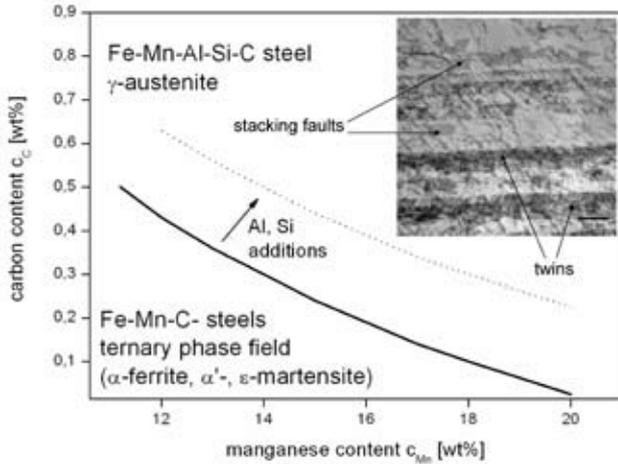
The research activities within the framework **Nanosopic Characterization of New Materials** is focused on a quantitative determination of phase separation on the nanometer scale and related hydrogen-induced embrittlement phenomena of high-alloy manganese steels (J. Deges). Another important topic performed in close collaboration with the Institute of Materials Physics, University of Göttingen, is the characterization of  $D0_3$  ordering, domain sizes and nanocrystalline structure by employing analytical TEM/EDS and high resolution field ion microscopy - APFIM and TAB - in  $Fe_3Al$  processed by severe plastic deformation via Equal Channel Angular Pressing (ECAP).

## Innovative Steel Research

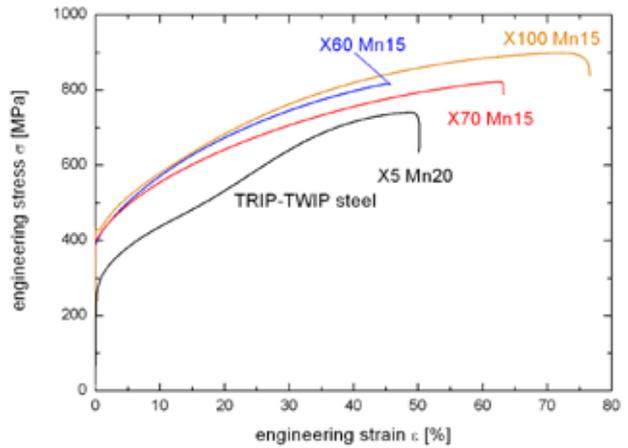
Steel, a classic metallic material, has been advanced in the last decade by considerable research and development efforts and progresses in metallurgical process technology into a high-tech structural and functional material. The versatile properties of steels and the high efficiency of steelmaking result in a growing worldwide demand for steel used in modern transportation systems and mechanical engineering constructions. These require high strength and high elastic stiffness as well as superior formability and fracture toughness. Another important aspect is the reduction in specific weight, materials and processing costs. The Department of Materials Technology has faced these great challenges and a variety of new classes of high manganese content light-weight steels have been developed and evaluated. The properties of these steels rely on specific deformation and strengthening mechanisms, such as multiple Transformation Induced Plasticity (m-TRIP), Twinning Induced Plasticity (TWIP) and Shear Band Induced Plasticity (SIP) effects causing high strength and superior ductility.

An interesting attempt is to create high-strength manganese steels which possess TRIP and TWIP behaviour in order to achieve optimum mechanical properties of both steel grades. A successful approach is the partly replacement of the high manganese content of TWIP steels by certain amounts of carbon. Therefore the stacking fault energy and the Gibbs energy can be systematically changed in order to determine the governing deformation and strengthening mechanisms. In addition carbon provides a considerable contribution to solid solution hardening of the iron-manganese austenite which increase the flow stress and tensile strength, respectively.

Fig. 1a illustrates the effects of manganese and carbon on the coexisting phase fields of Fe-Mn-C and Fe-Mn-Al-Si-C steels. It is obvious that lower carbon contents can replace larger amounts of manganese by keeping the austenite thermodynamically stable. Additions of Al and Si may shift the equilibrium boundary to larger amounts of carbon by keeping



**Fig. 1a:** Coexisting phase fields and related constituents of the Fe-Mn-C-diagram: Additions of Al and Si extend the ternary phase field. The inserted TEM micrograph reveals characteristic microstructural features like stacking faults and twins in  $\gamma$ -austenite of the high carbon Fe-Mn-Al-Si steels with TWIP behaviour.



**Fig. 1b:** Engineering stress-strain curves of diverse high manganese steels with different carbon contents revealing TRIP-TWIP and TWIP behaviour.

the manganese concentration constant. The inserted TEM bright field image reveals numerous stacking faults and deformation twins in high carbon content manganese steels with additions of Al and Si exhibiting TWIP behaviour.

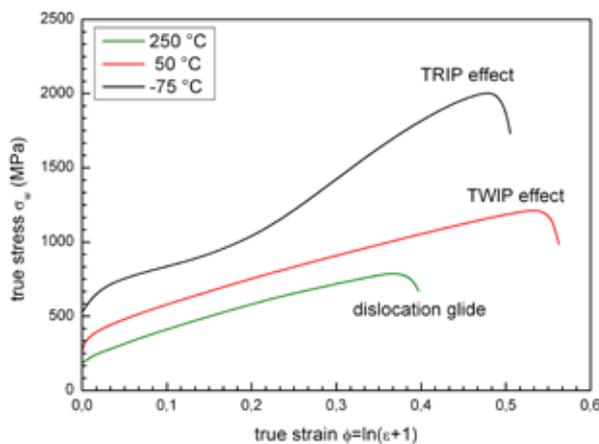
The engineering stress-strain curves in Fig. 1b show mixed TRIP-TWIP behaviour of a high manganese and low carbon steel possessing 20 wt.% Mn and characteristic TWIP behaviour of a high carbon content steel with 15 wt.% Mn. Each steel contains 2.5 wt.% Al and 2.5 wt.% Si.

Steadily increasing costs of the major alloying elements Cr, Ni and Mo to austenitic chromium nickel stainless steels has led to a concerted development of “low cost” stainless steels with reduced chromium and nickel contents exhibiting temperature and strain rate dependent TRIP and TWIP behaviour. These stainless steels reveal extensive deformation hardening accompanied by superior strength, fracture

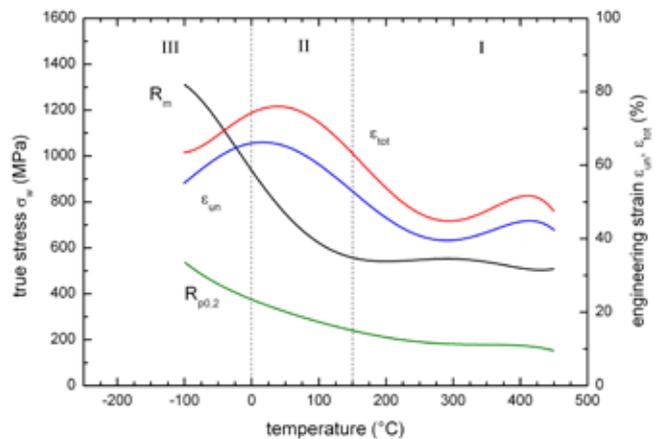
toughness and formability – deep drawing and stretch forming properties – in a wide temperature range from  $-100\text{ }^{\circ}\text{C} \leq \vartheta \leq 400\text{ }^{\circ}\text{C}$ . The true stress-strain diagram and the temperature dependent mechanical properties of a selected “low cost” stainless steel containing 3 % Ni, 16.5 % Cr, 8 % Mn and 0.25 % N-concentration in wt.%- are presented in Fig. 2a-b.

The three characteristic true stress vs. true strain curves in Fig. 2a and the related temperature regions I, II, III of Fig. 2b reveal the three principally acting deformation and strengthening mechanisms, such as the TRIP effect at temperatures of  $\leq -50\text{ }^{\circ}\text{C}$ , the TWIP effect in the temperature range of  $25\text{ }^{\circ}\text{C} \leq \vartheta \leq 150\text{ }^{\circ}\text{C}$  and the dominant dislocation glide process at  $\vartheta \geq 200\text{ }^{\circ}\text{C}$ . In the temperature transition regions overlapping mechanisms take place.

Due to the stress induced martensitic transformation of the austenite – TRIP effect – a strong deformation hardening occurs which causes the steep increase in



**Fig. 2a:** True stress-strain diagram exhibiting the temperature dependent TRIP and TWIP effects as well as the plastic deformation due to the dislocation glide in a modified “low cost” stainless steel.



**Fig. 2b:** True stress and engineering strain curves in dependence on the test temperature revealing three distinct regions related to the governing TRIP, TWIP and dislocation glide mechanisms.

the true stress-strain curve. Maximum tensile strength of about 1200 MPa and elongations to failure of 65 to 70 % have been achieved. The TWIP effect plays a dominant role when the tensile test is performed at room temperature up to 150 °C. This is characterized by an almost linear increase in the true stress-strain curve. An ultimate tensile strength of about 700 MPa and an elongation to failure of about 70 % have been recorded.

At deformation temperatures of  $\vartheta > 150$  °C the flow stress and tensile strength are decreasing and the conventional deformation and strengthening mechanisms due to dislocation glide and dislocation interactions will be dominant. However, the mechanical properties at these deformation temperatures are still improved in comparison with those of conventional austenite stainless steels tested at room temperature.

## Rapid Solidification Technology

Advanced processing technologies like new laser assisted welding and rapid prototyping as well as near net shape casting result in much higher cooling rates during solidification than conventional casting processes. The analysis and modelling of solidification in general and rapid solidification in particular are the main activities of the research group “rapid solidification technology” [1,2]. Another objective is the development of the corresponding process technologies and the related alloy development.

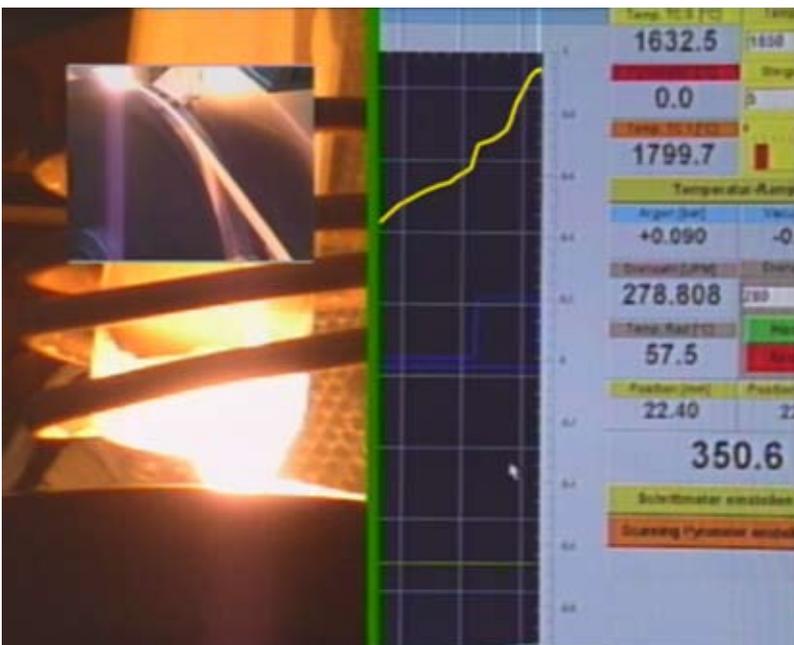
In order to understand and optimise processes involving high cooling rates, the measurement of relevant intrinsic parameters like the velocity of the solidification front is needed but experimentally not always accessible. Therefore, the calculation of solidification, phase and microstructure formation is one objective of the activities in this group. For a quantitative modelling the generalised en-

thalpy method is employed, which allows the computation of heat and mass transfer with multiple coexisting phases. The algorithms are embedded into superordinated FEM and FDM grids to simulate the correct boundary conditions [3]. In addition to these calculations, a planar flow casting (PFC) facility with a video recording system and a pyrometric scanner, which measures the temperature distribution along the longitudinal axis, is used to determine the kinetic parameters of different metallic melts (Fig. 3).

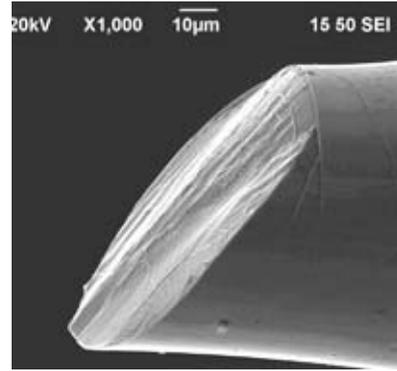
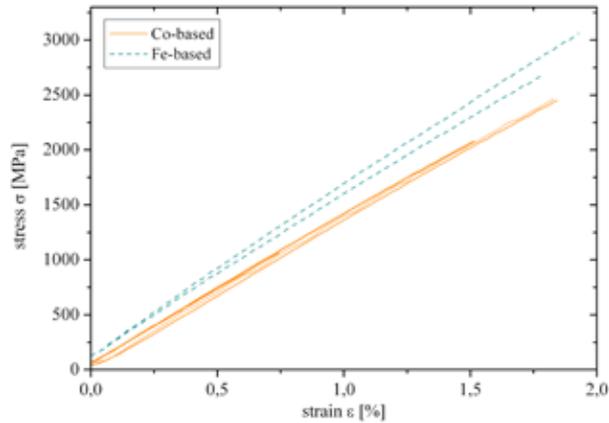
In addition to the PFC equipment the computer-controlled In-Rotating-Liquid-Spinning (INROLISP) facility is set up to produce thin wires in a single process step ranging from 50 to 150  $\mu\text{m}$  in diameter. The alloy is molten by an induction coil in an argon atmosphere and then injected into a laminar flowing coolant revealing a high cooling rate in the range of  $10^5$  K/s. Amorphous and nanocrystalline Fe(Co,Ni)SiB

wires are of great interest regarding their softmagnetic properties. Due to the amorphous morphology of the rapidly solidified material the magnetostriction is decreased and the wires are applied as sensor cores in Giant Magneto Impedance (GMI) sensors and high sensitive Procopiu sensors for micromagnetic detections [4].

Additional to softmagnetic applications, very high strength properties are achieved and correlated to the magnetic measurements under tensile strain. Yield stresses up to 3000 MPa at room temperature and Young’s moduli of about  $E \geq 150$  GPa are very promising for applications like reinforcing fibres. The wires reveal interesting strength, plasticity and fracture mechanism, as illustrated in the SEM image of Fig. 4. The plasticity results on shear bands induced by high local stresses on the surface of the wire.



**Fig. 3:** Recorded image taken from a PFC experiment showing the casting of a ribbon on a rotating wheel. On the side view (left) the crucible with the induction coil and the melt jet are visible. The front view (inserted image) shows the still glowing ribbon. The process parameters were recorded with a measurement program which is on the right half of the image.



**Fig. 4:** Representative stress-strain curves of ultrahigh strength Co- and Fe-based amorphous wires exhibiting tensile strength of the order of 2500 to 3000 MPa at room temperature at strain rates of  $10^{-5} \leq \dot{\epsilon} \leq 10^{-2} s^{-1}$ . The SEM image of the fracture surface of a Co-based wire reveals the vein pattern and traces of induced shear bands on the cylindrical wire surface.

The fracture of amorphous wires can occur either in a shear mode or in a brittle mode, depending on the constituent elements and on the binding energy which influences the rearrangements of the free volume and local shear processes. The investigation of the

brittle and ductile behaviour plays a key role in a profound understanding of the deformation kinetics in amorphous materials like shear band formation and viscous flow in the solid state [5].

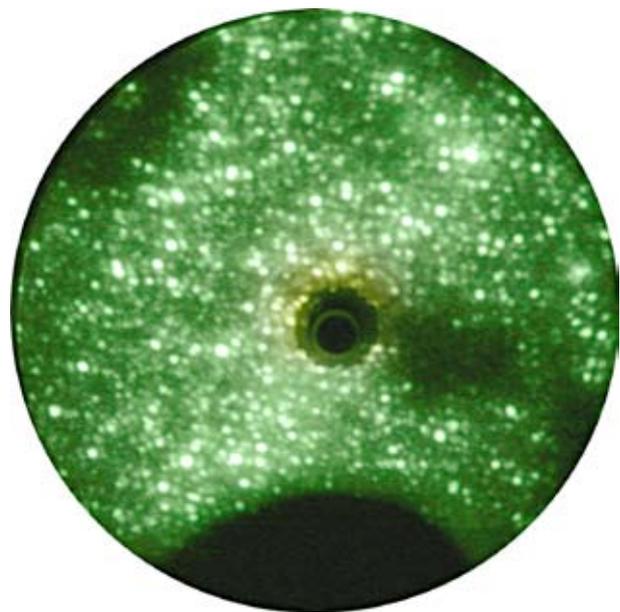
## Nanoscopic Characterization of New Materials

In the framework of mechanical and microstructural investigations on ultra high-strength austenitic steels with high manganese content, segregation of alloying elements has been observed on different length scales. Periodic structures of bright and dark contrast in TEM micrographs of some alloys indicate either the possible decomposition of the  $\gamma$ -matrix phase or a modulated elemental segregation. As the size of these brightly and darkly imaged areas in the structure is below the resolution of EDX- or even EELS analysis in the TEM and as XRD measurements display only one phase, atom probe field ion microscopic (APFIM) investigations have been performed.

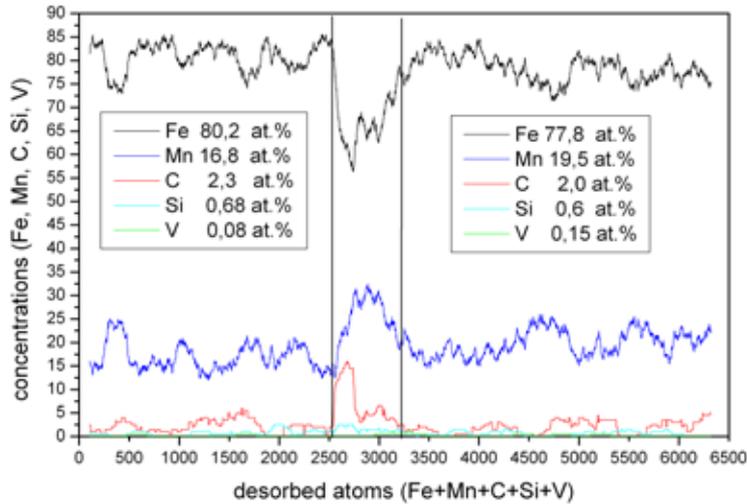
For the preparation of the APFIM specimen, a standard electrolytic polishing method has been used. The samples have been analysed with the APFIM in the Department of Materials Technology at a temperature of 70 K. Field ion image formation could only be observed at applied voltages above 11 kV. At lower voltages field evaporation of the surface atoms takes place before the image gas atoms are ionized.

The FIM image in Fig. 5 taken at  $V_{tip} = 15.8$  kV and an image gas (neon) pressure of  $p_{Ne} = 4 \times 10^{-5}$  mbar shows hardly any crystallographic poles which is typical for high-alloy steels. Therefore it was not possible to determine the crystallographic orientations in the APFIM analysis. On the other hand some brightly and darkly imaged regions on the FIM micrograph can be distinguished which are, if they

are not connected with crystallographic poles, an indication for different compositions (the dark feature in the lower part of the image is an obstacle between the image screen and the camera). This interpretation is supported by atom probe measurements as it is shown in the concentration profile in Fig. 6. Two regions with slightly different compositions can be clearly distinguished which are separated by a



**Fig. 5:** FIM-image of a high manganese steel showing regions with different brightness indicating concentration fluctuations.



**Fig. 6:** Concentration profile of a high manganese steel exhibiting two regions with different composition separated by a zone of co-segregated C and Mn.

small zone of strong co-segregation of carbon and manganese atoms. The apparent width of this zone is approximately 3 nm which can also be explained if a sharp interface is non-perpendicularly cut by the atom probe measurement.

To our knowledge this is the first atom probe investigation on this class of high manganese austenitic steels and the results strongly support other investigations on modulated structures and phase separation in the Fe-Mn system.

## Ordered Alloys for High-Temperature Applications

Alloys with superlattices exhibit extraordinary physical, chemical and mechanical properties due to their specific electronic structures or magnetic states, packaging densities and chemical bonds of the coexisting atoms. Technical iron or nickel base alloys and steels possess a large variety of ordered compounds. One of the most prominent among others is the hard  $\sigma$ -phase (FeCr) with complex tetragonal crystal structure of the iron-chromium system occurring in duplex stainless steels or in high-alloyed austenitic chromium-nickel stainless steels after thermal exposure. In hardmagnetic iron-cobalt alloys with additions of vanadium and chromium the B2-orderd FeCo solid solution reveals a relatively high energy product and a maximum magnetic saturation. The nanocrystalline  $\Phi$ -phase  $\text{Nd}_2\text{Fe}_{14}\text{B}$  with complex tetragonal type of structure embedded in an iron matrix exhibits the maximum energy product and extremely high coercive forces in comparison to any other magnetic materials.

In nickel-based superalloys, the most favorable strengthener is the intermetallic  $\text{Ni}_3\text{Al}$  compound  $\gamma'$ -phase- which precipitates out in a  $\gamma$ -nickel solid solution matrix and increases the warm strength and high-temperature creep resistance as well.

The aim of the research initiative on ordered alloys is the fundamental study of their constitutions and

the development of creep and oxidation resistant high-temperature materials, based on iron-, cobalt-, nickel and titanium light-weight aluminides in order to reduce the fuel consumption, the emission of exhaust gases and to improve the thermal efficiency of advanced energy conversion systems, such as stationary gas turbines of power plants, jet engines of the aerospace industry and diesel or gasoline engines of the automotive industries.

A sound knowledge of the constitution of alloy systems is the basis for any aimed alloy development. Therefore phase equilibria and phase transformations have been experimentally studied within various systems. In a joint project (Innovative Materials Group, Universität Wien; Joining and Welding Research Institute, Osaka University; MPIE) the constitution of the Fe-Si-Ti system has been investigated in the temperature range 900 and 1700 °C [6]. Based on light optical microscopy (LOM), scanning electron microscopy (SEM), powder X-ray diffraction (XRD), electron probe microanalysis (EPMA), and differential thermal analysis (DTA) on alloys of 80 different compositions, nine ternary compounds were characterised and an isothermal section at 900 °C, a liquidus projection and a reaction scheme were established. In co-operation with MPI-CPfS (Dresden) the Co-Nb system has been reinvestigated [7]. By



LOM, XRD, EPMA and DTA on equilibrated samples of fixed compositions and additional investigations on a number of diffusion couples phase equilibria with special emphasis on the homogeneity ranges of the three Laves phases that are stable within this system have been determined. Together with EMPA (Dübendorf, Switzerland) phase equilibria within the Fe-Ni-Ti system were investigated which resulted in two isothermal sections for 800 and 1000 °C, respectively [8].

Because Fe-Al-based materials are an inter-departmental research topic at MPIE a number of respective systems have been studied including reinvestigation of the transition temperatures in the binary Fe-Al system [9]. Besides an accurate determination of the liquidus and solidus temperatures in the Fe-rich part of the Fe-Al system, the temperature for the transition from ordered B2 to disordered A2 was substantially corrected. In order to evaluate the possibility of strengthening Fe-Al-based alloys through precipitates, the Fe-Al-Mo [10,11] and Fe-Al-Nb [12] systems have been studied. Isothermal sections established at 800, 1000 and 1150 °C for the Fe-Al-Mo system reveal that the solid solubility for Mo in  $\alpha(\text{Fe,Al})/\text{FeAl}$  decreases markedly from about 19 at.% at 1150 °C to about 6 at.% at 800 °C, i.e. giving the possibility to generate fine-scaled precipitates of  $\text{Fe}_7\text{Mo}_6$  or  $\text{AlMo}_3$  in the Fe-Al matrix during cooling. In contrast, the solid solubility for Nb in Fe-Al varies only little with temperature thus allowing generating two-phase microstructures with the Laves phase which are stable within a wide range of temperatures. Such microstructures have been generated by directionally solidification and their mechanical properties have been established [13]. The aligned microstructures showed a high stability during creep, i.e. no coarsening was observed after long-term creep at 750 °C. Because vacancies and minor amounts of carbon have a marked influence on the properties of Fe-Al-based alloys, the latter effect was studied in a joint project by the Physics of Metals and Materials Science Dept. (Tula State University, Russia), Institute for Materials (TU Braunschweig), and MPIE by mechanical spectroscopy in Fe-Al-Si alloys [14]. Finally, the complex constitution and structural variations of the Heusler phase  $\text{Ti}_{25}(\text{Fe}_{50-x}\text{Ni}_x)\text{Al}_{25}$  ( $0 \leq x \leq 50$ ) have been investigated by EPMA, XRD and in-situ neutron powder diffraction in a collaborative project by Institute of Physical Chemistry (Universität Wien), Laboratory for Neutron Scattering (ETH Zurich), Paul Scherrer Institut (Villingen), and MPIE [15].

The above mentioned Heusler phase plays an important role in the development of Fe-Al-Ti-based alloys with drastically improved creep resistance [16,17]. The mechanical properties and corrosion behaviour of such alloys modified by adding Cr and B in order to increase ductility at elevated temperatures

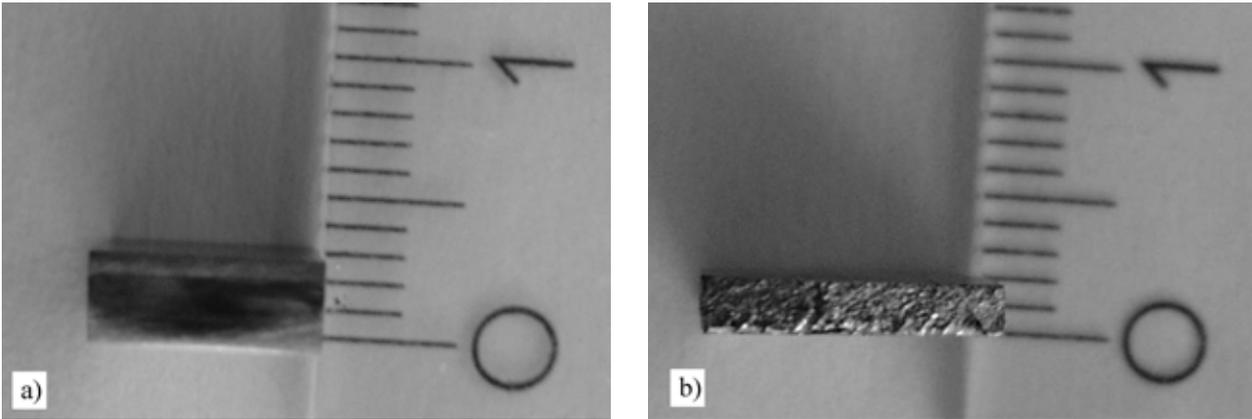
has been studied in detail. In these studies it was found that Fe-Al-Ti-based alloys show a two-fold flow stress anomaly [18], an unusual feature which has not been reported before. The achievements made in the development of Fe-Al-based alloys through the various interdepartmental research projects at MPIE have been summarised in [16,19].

Al-rich TiAl alloys have an even lower density and much improved corrosion resistance compared to “conventional” Ti-rich  $\gamma$ -TiAl alloys. It has been found at MPIE that also in Al-rich TiAl alloys lamellar microstructures can be generated. As such microstructures are essential for the properties of Ti-rich  $\gamma$ -TiAl alloys a large scale joint project between the University of Magdeburg, ACCESS e.V. (Aachen), Caesar (Bonn), Christian-Albrechts Universität (Kiel), and MPIE funded by the DFG has been initiated. Within this project the kinetics of the formation of lamellar  $\gamma$ -TiAl +  $\text{Al}_2\text{Ti}$  microstructures and the dissolution of metastable  $\text{Al}_5\text{Ti}_3$  are studied [20] and production of alloys by different casting techniques and their mechanical properties are evaluated [21].

Within the inter-institutional research initiative “The Nature of Laves Phases”, which is described in detail on page 22 in Part I of this report, currently four different research projects on Laves phases are in progress at the MPIE. The first project deals with the ternary systems Nb-TM-Al (TM = Cr, Fe, Co) which have extended phase fields of the cubic C15 and the hexagonal C14 Laves phases. Isothermal sections of the ternary phase diagrams are established by detailed experimental investigations of equilibrated alloys heat-treated at temperatures ranging from 800 to 1450°C. Site occupancies of the different atom types in the ternary C14 Laves phase lattice are studied by Rietveld refinements of powder XRD patterns in order to discuss the stability of the hexagonal vs. cubic crystal structures [22].

In a parallel, more recently started project, thermodynamic modelling is performed on the above mentioned ternary systems by the CALPHAD method. Based on the experimentally obtained phase equilibria and invariant reaction temperatures, isothermal sections of the ternary phase diagrams can be calculated for experimentally inaccessible temperatures enabling a detailed discussion of stability and transformation of the different Laves phase polytypes [23].

The mechanical properties of Laves phases are investigated in dependence on structure and composition in another project of this research initiative. Deviations from the stoichiometric composition result in defects which are anti site atoms in case of transition metal Laves phases. It is well-known for metals and many intermetallic phases that the introduction of defects in the crystal structure results in a hardening of the material. From microhardness



**Fig. 7:** Rectangular block of the intermetallic phase  $Nb_2Co_7$ , before (a) and after (b) hammering at room temperature showing unusual, 'pseudo-ductile' behaviour (the scale is in cm).

measurements on single-phase Laves phase alloys and nanoindentation scans along concentration profiles in diffusion couples containing Laves phases, the surprising observation of defect softening was made for Laves phases with compositions deviating from the stoichiometry. To find an explanation of this effect and its relation to the only partly understood mechanisms of plastic deformation in Laves phase structures is one goal of the project [24].

In a fourth project, the phase relations of Laves phases in the binary Cr-Ti system are experimentally investigated. Three different polytypes of Laves phases (C14, C15 and C36) are reported to exist in this system. Using equilibrated alloys as well as Cr-Ti diffusion couples, the stability and homogeneity ranges of these phases are studied in order to understand the complicated co-existence of these phases [25].

In the Co-rich part of the binary system Co-Nb, the intermetallic phase  $Nb_2Co_7$  is formed in a peritectoid reaction from the  $Co(Nb)$  solid solution and the Laves phase  $NbCo_2$ .  $Nb_2Co_7$  possesses a monoclinic crystal structure with details of it resembling the crystal structure of Laves phases [26,27]. Single-phase alloys of this monoclinic intermetallic phase show an unusual mechanical behaviour. The material can be strongly deformed at room temperature by beating with a hammer without shattering or fracturing and can be strained in compression to at least 5% without fracture (Fig. 7). Nevertheless,  $Nb_2Co_7$  behaves like a brittle material when tested under bending or tensile load. The mechanical properties of this phase were studied by microhardness measurements as well as compression, tensile and bending tests. The microstructure was analysed by light-optical and scanning electron microscopy prior to and after testing [27,28]. Within a co-operation with Prof. S. Kumar from Brown University (Providence, RI, USA), the microstructural evolution and phase transformations

in as-cast, two-phase Co-15at.%Nb alloys are studied by in-situ transmission electron microscopy. During annealing the sample at temperatures between 700 and 1000°C, the formation of the  $Nb_2Co_7$  phase from the fine, lamellar two-phase Co + Laves phase microstructure can be observed. The formation process of the  $Nb_2Co_7$  phase is very slow, whereas its decomposition after heating above the peritectoid reaction temperature is a fast process. DTA investigations are performed to study the kinetics of this peritectoid reaction [29].

The elastic properties, specifically the bulk moduli, and the volume expansion of a variety of intermetallics which rank among the types of Hume-Rothery, Zintl or interstitial compounds, such as the aluminides: NiAl (B2),  $Ni_3Al$  ( $L1_2$ ),  $NbAl_3$  ( $D0_{22}$ ), and silicides:  $Mg_2Si$  (C1),  $NbSi_2$  (C40),  $CrSi_2$  (C40),  $TiSi_2$  (C54),  $ZrSi_2$  (C49)  $MoSi_2$  ( $11_b$ ) and  $WSi_2$  ( $11_b$ ) have been investigated with respect to their lattice structures -space groups-, binding energies and atomic volumes. The work was carried out in close collaboration with the Institute für Geowissenschaften und Lithosphärenforschung, Justus-Liebig-Universität Gießen. The measurements have been performed by using the high pressure facility Multi-Anvil X-ray source -MAX 80-, provided by synchrotron radiation at HASYLAB, DESY. The maximum applied pressure was 6 GPa and the test temperature ranged from 25 °C up to 1200 °C. The recorded changes in the lattice parameters and in the unit cell volumes of the intermetallics were analysed by applying the Birch-Murnaghan equation of state for determining the bulk moduli. The volume expansion as a function of the temperature was derived by using the Helmholtz free energy concept in combination with the  $\Delta V$ -T relation. From this the thermodynamic Grüneisen-parameters of the compounds have been determined. The bulk moduli of the intermetallic compounds are related to their lattice energies and atomic volumes [30].



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## Research Projects in Progress

### Innovative Steel Research

*Bausch, Rablbauer, Frommeyer:* Industrial up-scaling of Mn-Al-C steel metallurgy

*Bausch, Rablbauer, Frommeyer:* Investigations on the plastic deformation and strengthening mechanisms of carbide particle strengthened Mn-Al-C steels

*Deges, Frommeyer, Rablbauer, Rademacher, Al-Kassab:* Processing, microstructural investigation and mechanical properties determination of ultra-fine-grained and nanocrystalline ordered Fe<sub>3</sub>Al-(X=Cr, Ti) alloys

*Deges, Frommeyer, Rablbauer:* Mechanical and physical properties of ternary Fe<sub>3</sub>Al-Me (Me = Ti, V, Nb, Ta, Cr, Mo, W, Mn, Co, Ni, Cu, Si) alloys with D0<sub>3</sub> ordered structure

*Frommeyer, Brokmeier, Rablbauer:* Structural investigations on Super TRIP steels

*Frommeyer, Brokmeier:* Development and evaluation of quasi eutectoid high carbon-aluminium steels

*Frommeyer:* Investigations on the mechanism of martensitic transformations in iron carbon steels

*Frommeyer, Kaiser, Rablbauer*: Development of heat resistant steels for high temperature application

*Maier, Rablbauer, Frommeyer*: Investigations on plastic deformation behaviour in TRIP steels

*Maier, Rablbauer, Frommeyer*: Plastic deformation behaviour of austenitic steels under multi-axial loading

*Rablbauer, Frommeyer*: Development and characterisation of ferritic stainless Al-Cr steels of deep drawing quality

*Rablbauer, Buchwalter, Deges, Frommeyer*: Development and Characterisation of ultrahigh strength perlitic steel wires

*Rablbauer, Georgeou, Frommeyer*: Development of FeAl base alloys for power train application in automotive

*Stein, Frommeyer*: Soft-magnetic Fe-Si and Fe-Si-Al alloys processed under microgravity

### Rapid Solidification Technology

*Frommeyer*: Development of alloying systems for flux free soldering of aluminium alloys

*Rablbauer, Wenke*: Corrosion resistant alloy development with optimised heat conductivity and thermal expansion coefficient for processes with high cooling rates

*Wenke*: Investigations on melt pool undercooling of aluminium alloys during the meltspinning process

*Wenke*: Numerical meltspinning simulations of phase and microstructure formation of aluminium alloys under high cooling rates

*Wenke*: Modelling of phase and microstructure formation under non-equilibrium conditions of aluminium-titanium laser welded seams

*Zeller*: Continuous casting of soft magnetic fibres for new magnetic field sensors

*Zeller*: Investigations on continuously cast fibres and wires of Cu-base shape memory alloys

*Zeller, Wenke*: Casting of thin Co-base alloy ribbons under atmospheric conditions with the meltspinning process

### Development and Characterisation of New Materials

*Engberding, Palm, Stein*: Transformation of h-Al<sub>2</sub>Ti and Al<sub>5</sub>Ti<sub>3</sub> and the formation of lamellar TiAl + r-Al<sub>2</sub>Ti microstructures

*He, Palm, Stein*: CALPHAD assessments of the systems Co-Nb, Al-Co, Al-Co-Nb, and Al-Cr-Nb

*He, Palm, Stein, Schuster\** (\*University of Vienna): Investigation of the liquidus surface of the Al-Co-Nb system

*Krein, Palm, Janschek\*, Baur-Partenheimer\** (\*Leis- tritz Turbinenkomponenten Remscheid GmbH), *Hanus\*\** (\*\* Technical University Liberec): Forging of steam turbine blades from an advanced Fe<sub>3</sub>Al-based alloy

*Palm, Hanus\** (\*Technical University Liberec, Czech Republic): Phase equilibria in the Fe-Al-Ta system

*Palm, Hotar\** (\*Technical University Liberec, Czech Republic): Oxidation behaviour of Fe-Al-Ta alloys

*Palm, Hotar\** (\*Technical University Liberec, Czech Republic): Corrosion of Fe-Al-base alloys in water vapour

*Palm, Stein, Engberding, Heilmaier\*, Saage\*, Sturm\** (\*Univ. Magdeburg), *Drevermann\*\*, Schmitz\*\*, Aguilar\*\** (\*\*Access eV, Aachen), *Irsen\*\*\** (\*\*caesar, Bonn), *Kienle\*\*\*\** (\*\*\*\*Christian-Albrechts-Univ. Kiel): Al-rich AlTi alloys

*Prymak, Stein*: Phase equilibria in the Nb-Al-X (X = Cr, Fe) systems

*Prymak, Stein*: Rietveld refinement of ternary C14 Laves phase crystal structures

*Stein*: DTA investigations of the kinetics of peritectoid phase transformations

*Stein, Frommeyer*: Microstructure and mechanical behaviour of Zr- and B-doped monoclinic Nb<sub>2</sub>Co<sub>7</sub>

*Stein, Ishikawa\** (\*Tokyo Institute of Technology, Tokyo, Japan): Experimental investigation of the Cr-Ti system

*Stein, Palm, Kumar\** (\*Brown Univ., Providence, RI, USA): In-situ electron microscopy studies of micro- structural evolution of Co-Nb Laves phase alloys

*Stein, Palm, Raabe, Frommeyer, Kreiner\*, Grin\** (\*MPI-CPfS, Dresden), *\*\*Leineweber, \*\*Mittemeijer* (\*\*MPI-MF, Stuttgart), *Fischer\*\*\*, Jansen\*\*\** (\*\*MPI-FKF, Stuttgart): MPG Research Initiative: The nature of Laves phases

*Stein, Palm, Vogel\** (\*LANL, Los Alamos, NM, USA): Investigation of crystal structures of Fe-Al(-Mo) inter- metallic compounds at high temperatures by in-situ neutron diffraction

*Stein, Rablbauer, Frommeyer*: Solidification of NiAl-X (X = Cr, Re) alloys under microgravity

*Voß, Palm, Stein*: Mechanical properties of transition metal Laves phases



# Department of Microstructure Physics and Metal Forming

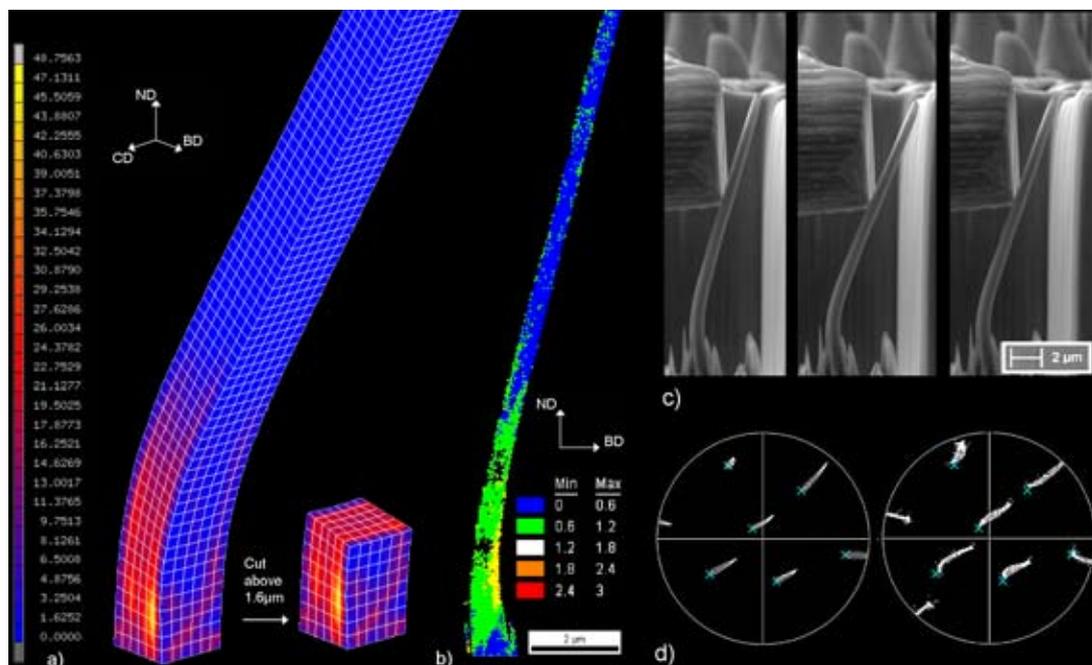
D. Raabe

## Scientific Concepts

The department conducts basic research on the mechanical properties of crystalline materials. These are determined by the thermodynamic constitution of the material on the one hand and the kinetics of microstructure evolution on the other hand. Microstructure evolves in crystalline matter primarily through elasto-plastic deformation, phase transformations (typically non-equilibrium), recrystallization, and grain growth so that studies on the underlying mechanisms of these processes form the basis of our work.

In the case of engineering materials such as steel, titanium, magnesium, and nickel-base alloys microstructures evolve under complex thermomechanical history and boundary conditions. This means that the effect of processing on the microstructure and hence on the final behaviour of the materials is taken into consideration in our projects.

In order to elucidate which specific mechanisms lead to certain microstructures we use a combination of advanced characterization, well defined model experiments, and theoretical models of microstructure evolution and microstructure-property relationships at different length scales, Fig. 1. A close integration of theory and experiment is, hence, a common feature of our approach [1-8]. Most of the microstructure models that are developed in our department are formulated as continuum-based rate formulations of lattice defect kinetics and corresponding kinetic equations of state describing the resulting properties. The underlying differential equations are typically solved using variational methods [9-16]. Corresponding simulations are then conducted under boundary conditions that are of interest with respect to a certain processing step or model experiment. Typical examples of such models are dislocation-density based crystal plasticity formulations that are solved by using standard finite element solvers [4-16].



**Fig. 1:** Experiment, analysis, and crystal plasticity finite element simulation of bending of a copper nanowire single crystal. a): Crystal plasticity finite element simulation of the distribution of the density of geometrically necessary dislocations [ $1/\mu\text{m}^2$ ] together with sectional view (the nanowire was cropped above  $1.6\mu\text{m}$ ); b): Experiment: average local lattice misorientation distribution (Kernel average misorientation) obtained from high resolution EBSD data. The lattice misorientation is a measure for the density of geometrically necessary dislocations (colour coding in  $^\circ$ , maximum:  $3^\circ$ ). Note that not the complete cross section could be measured. c): Sequence of 3 subsequent bending steps; d) pole figures obtained from simulation (left) and experiment (right). CD: Cross direction, BD: Bending direction, ND: Normal direction [1]. The work was a part of the Bachelor thesis of F. Weber.

We use simulations not only for studying the fundamentals of microstructure evolution and the resulting properties: Owing to the increasing complexity of modern mechanical experimental and characterization results, particularly at nanoscopic scales, it is often difficult to interpret experimental observations without the use of accompanying simulations. Often microstructure observations can be highly sensitive with respect both to the boundary conditions imposed

during an experiment and also to the underlying mechanisms that take place before or during an experiment [1-3,5]. In such cases simulations help us to critically interrogate the true relationships between microstructure mechanisms and boundary conditions imposed during an experiment. In other words, the inverse use of simulations can help us to identify what the true boundary conditions of an experiment or process have been, Fig. 1 [1].

## Scientific Groups and Interdisciplinary Research

### Overview

For pursuing the aims outlined above six main competence groups are currently operative. They form the cornerstones of the department.

- Theory and Simulation (F. Roters)
- Microscopy and Diffraction (S. Zaefferer)
- Thermomechanical Processing (D. Ponge)
- Biological Materials (S. Hild)

- MPG-FhG-Group on Computational Mechanics of Polycrystals (P. Eisenlohr)
- MMM-Group on the Theory and Simulation of Complex Fluids (F. Varnik)

The latter two initiatives are temporary groups. The project of P. Eisenlohr on the Computational Mechanics of Polycrystals is jointly funded after two subsequent evaluation workshops (2005, 2008) by the Max-Planck-Society and the Fraunhofer-Society

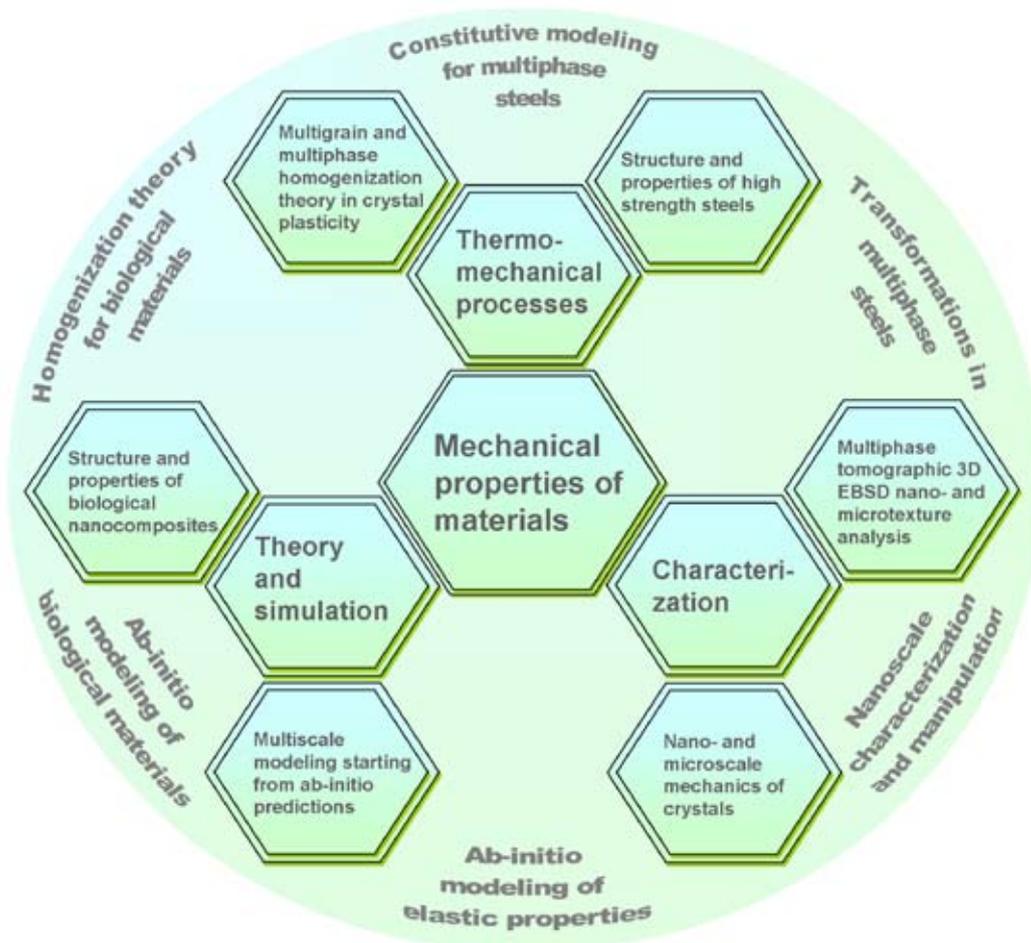


Fig. 2: Connection between the main research areas in the department and examples of corresponding projects.



for 3+3 years, 2005-2011. The project of F. Varnik on the Theory and Simulation of Complex Fluids is funded by the Max-Planck-Multiscale Modelling Initiative for 4 years, 2005-2009.

Many projects in our department are jointly pursued by members of different groups including also other departments. The most important interdisciplinary research fields of the department during the past two years are given in the list below. Fig. 2 presents the connection between these research areas and gives examples of corresponding projects.

- Structure and properties of high strength steels
- Multiphase tomographic 3D EBSD nano- and microtexture analysis
- Nano- and microscale mechanics of crystals
- Multiscale modelling starting from ab-initio predictions
- Structure and properties of biological nano-composites
- Multigrain and multiphase homogenization theory in crystal plasticity

The projects are conducted using a number of highly specialized laboratories. The main laboratory facilities of the department are

- Mechanical testing laboratory
- High resolution scanning orientation electron microscopy
- 3D electron microscopy: Joint focused ion beam microscopy and 3D nanotexture laboratory
- Transmission electron microscopy laboratory
- X-ray diffraction laboratory
- Strain mapping digital image correlation (photogrammetry) laboratory
- Surface confocal topography laboratory
- Nanomechanical testing and atomic force microscopy laboratory
- Laboratory for deformation dilatometry
- Computational materials science laboratory
- Large scale thermomechanical treatment laboratory
- Hot and cold rolling laboratory
- Heat treatment laboratory
- Laboratory for mechanical testing
- Micromechanical testing laboratory
- Optical metallography laboratory

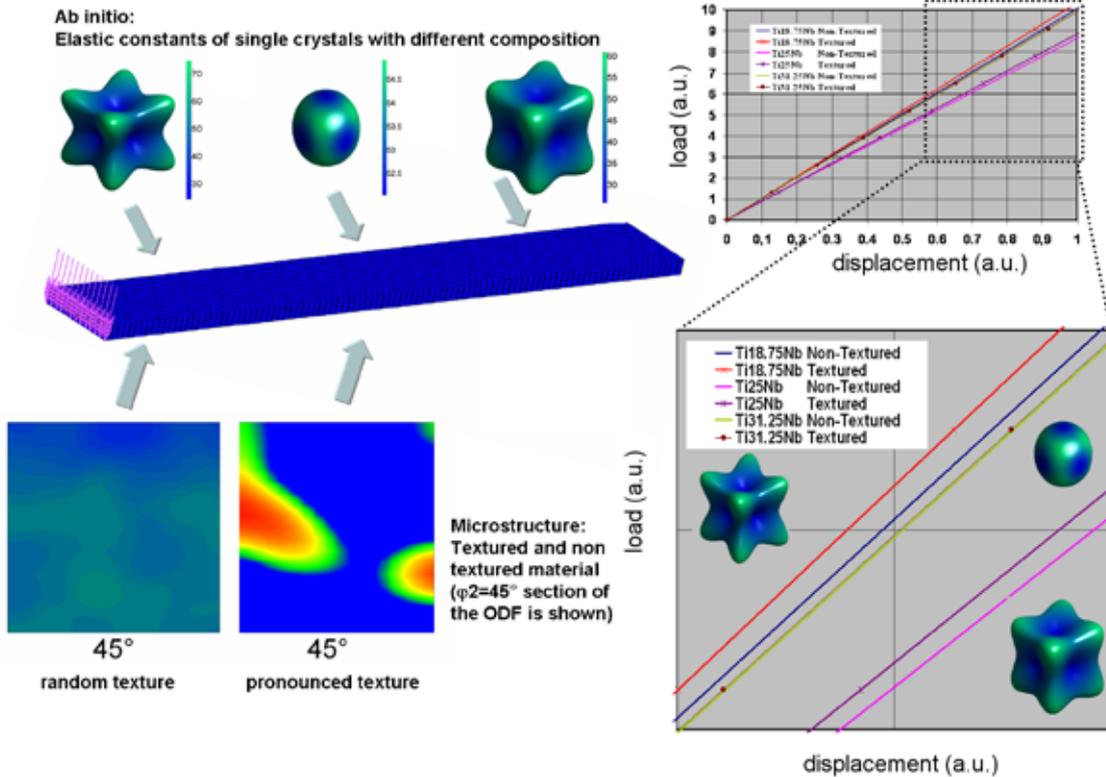
In the following sections we present the scientific directions of the different groups and give examples of their projects and interdisciplinary cooperations.

### Theory and Simulation (F. Roters)

#### Group Mission and Interdisciplinary Activities.

The group for Theory and Simulation develops physics-based crystal plasticity constitutive models for the micromechanical behaviour of crystalline materials [1-8]. The models are based on rate formulations that describe the evolution of lattice defects under external loads [9-16]. The constitutive rate formulations account for the crystalline nature of the material, i.e. they describe lattice defect kinetics individually on all crystallographic shear systems. This approach renders the constitutive models anisotropic. The formulations are built on mobile, sessile, and geometrically necessary dislocation populations (Fig. 1), and on mechanical twinning as underlying deformation carriers. The resulting lattice defect density quantities are used in kinematic state equations for the calculation of the local and global stresses. The mechanics at grain boundaries and heterophase interfaces are also addressed. Standard nonlinear Finite Element solvers (FEM) are used for solving the coupled sets of partial differential equations rendering the approach an internal-variable based crystal plasticity finite element method [10-16].

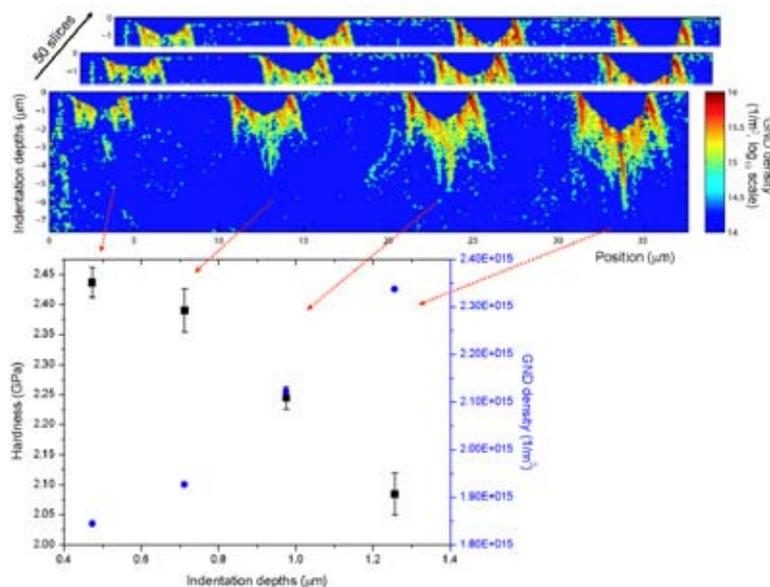
Recently, particular attention has been placed on advanced multi-scale crystal plasticity finite element simulations. Three directions are pursued in this context. The first approach lies in an increasing interest to consider the (tensorial) stress fields of single dislocations in crystal plasticity constitutive models. In some cases such as Bauschinger phenomena (flow stress changes upon load reversal) the effect of the tensor stress field of individual dislocations seems to play an important role for micro- and nanoscale deformation problems. The second field concerns the direct use ab initio simulation results in crystal plasticity finite element simulations, Fig. 3 [17-19]. Typical examples are the use of phase fractions and / or elastic constants that cannot be obtained by experiment as input parameters in crystal mechanical simulations. These projects are pursued in close cooperation with the Department for Computational Materials Design (Prof. Neugebauer). The third direction is the development of advanced multiphase and polycrystal homogenization models to render crystal mechanical simulations also suited for large scale problems. The latter aspect will be discussed in more detail below in the context of the Max-Planck-Fraunhofer Group on Computational Mechanics of Polycrystals (P. Eisenlohr) and also in form of a dedicated short article, see p. 221 of this volume.



**Fig. 3:** Example of the combination of *ab initio* input data and crystal mechanical finite element simulations. The current case is an example of using elastic tensor data of single crystals obtained for body centered cubic Ti-Nb alloys of different composition by DFT-based *ab initio* simulations. The data are used to simulate the elastic stiffness during bending of randomly and non-randomly textured polycrystals. The project is part of the ongoing Ph.D. thesis of D. Ma. The project is conducted in close cooperation with the group of Dr. Friak in the Department for Computational Materials Design (Prof. Neugebauer).

**Highlights 2007-2008.** During the past 2 years the group Theory and Simulation has mainly concentrated on the development of crystal plasticity models that consider the formation of deformation-induced twins and their interaction with the microstructure,

particularly with the lattice dislocations and internal interfaces. The approach is built on the local stress-assisted nucleation of twins and their expansion during deformation. Better understanding and modelling of these mechanisms is essential for optimal design



**Fig. 4:** Joint analysis of the indentation hardness for 4 indents. The total GND density below each indent is shown (blue symbols) together with the measured hardness values (black symbols). The GND density is obtained by summing the GNDs over all 50 individual 2D EBSD sections and dividing this number by the total reference volume. The data show that the GND density decreases with decreasing indentation depth and increasing hardness. The density of the GNDs decreases faster with decreasing indentation depth than the affected reference volume. This leads to an overall decrease in the GND density for smaller indents. The project is part of the Ph.D. thesis of E. Demir. It is based on the close cooperation of the groups of Dr. Roters and Dr. Zaerrerer.



of advanced TWIP-assisted high strength steels (TWIP: twinning induced plasticity). These steels yield very high flow stresses together with excellent formability. This combination is due to the fact that intense deformation twinning in these novel steels takes place at strain levels when localization tends to occur. The additional strain hardening resulting from twinning compensates for geometrical softening and leads to very large deformations.

Another challenge lies in the development of advanced dislocation based constitutive laws for applications at very small crystal plasticity scales (nm- $\mu\text{m}$ ) where deformation gradients, dislocation source depletion, and intricate contact mechanisms can dominate the deformation, Fig. 1 [1-3]. For example Fig. 4 shows a study on the link between the indentation size effect and the density of geometrically necessary dislocations (GNDs): Four indents of different depth and hardness were placed in Cu (conical indenter). The deformation-induced lattice rotations below the indents were monitored via 3D EBSD. From these data we calculated the first order gradients of strain and the GND densities below the indents. This approach help to quantify both the mechanical parameters (depth, hardness) and the lattice defects (GNDs) that are held responsible for the indentation size effect. It was observed that the GND density does not increase with decreasing indentation depth but it drops instead.

### Microscopy and Diffraction (S. Zaefferer)

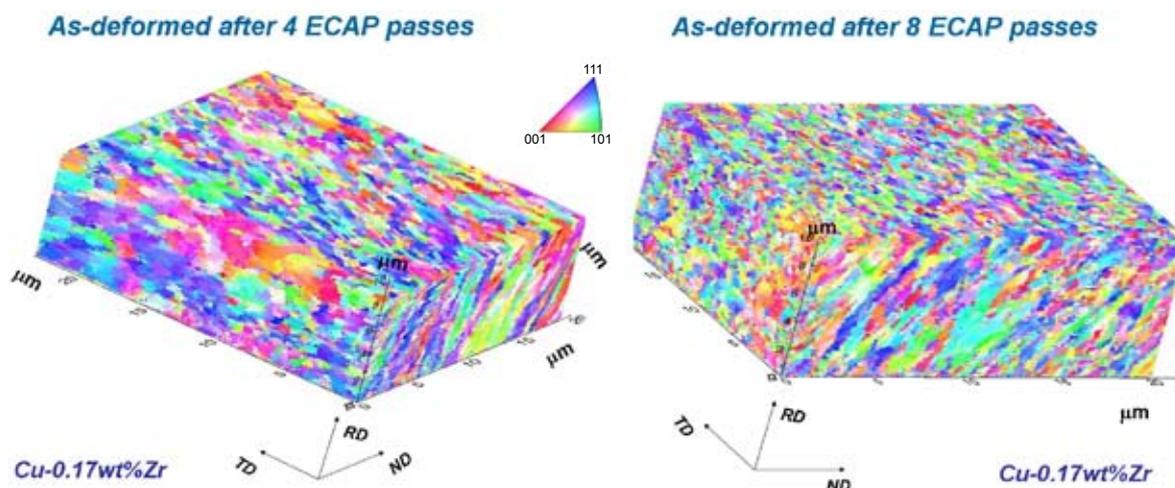
#### Group Mission and Interdisciplinary Activities.

The aim of the Microscopy and Diffraction group is two-fold: on the one hand the group deals with the experimental investigation of microstructure formation mechanisms, including basic plasticity, phase transformation, and recrystallization mechanisms in

complex engineering multiphase materials [20-28]. On the other hand the group develops new tools and techniques for corresponding investigations, particularly such based on orientation microscopy in the SEM and the TEM but also x-ray based techniques [27-30]. Concerning the investigation part special emphasis is placed on the more precise observation and understanding of athermal transformation mechanisms and the proper characterization of the interfaces involved. Regarding recrystallization phenomena projects are particularly concerned with nucleation phenomena and grain growth. These mechanisms play key roles in understanding the kinetics of microstructure evolution and structure-property relations in modern steels. For instance modern TWIP, TRIP, and dual phase steels (TRIP: transformation induced plasticity) can only be understood on the basis of a detailed characterization of the different bainite, martensite, austenite, ferrite, carbide, and intermetallic phases on all relevant length scale ranging from nm to mm.

Owing to the small size of many of these microstructural features and the necessity for their crystallographic characterization the group employs mainly electron optical and diffraction techniques. Key development projects are currently the 3-dimensional EBSD-based orientation microscopy (Fig. 5), highest resolution EBSD (with a spatial resolution below 10 nm), and fully automated TEM-based orientation microscopy.

A further important project concerns the development of microscopy techniques for the in-situ observation of damage initiation mechanisms in crystalline materials. The group is equipped with a variety of advanced instruments for microstructure and texture characterization. Among these is a high-resolution Zeiss Crossbeam XB1560 FIB for 3D EBSD investigations. This machine is a combination



**Fig. 5:** 3D EBSD analysis of a Cu-0.17wt%Zr sample after 4 and 8 ECAP passes, respectively (ECAP: equal channel angular pressing), as an example for advanced 3D microstructure characterization. The measurements were performed by A. Khorashadzadeh in an ongoing Ph.D. thesis. The project is funded by a DFG research group (Forschergruppe 544) on the Mechanical Properties and Interfaces of Ultra Fine Grained Materials. The EBSD step size was 150 nm and the distance between the slices was 150 nm.

of a high resolution SEM and a focussed ion beam (FIB) microscope equipped with a TSL EBSD system and EDX. For 2D EBSD investigations, a high-resolution, high-beam SEM (JEOL JSM 6500 F) with a TSL EBSD system as well as a standard tungsten filament SEM (JEOL JSM 840A) with an HKL Technology EBSD system are available. All types of SEM instruments allow one to mount micro-tension machines of different dimensions for in-situ deformation tests. A heating stage is also available for in situ transformation experiments. For TEM a Phillips CM 20 is used. This instrument is equipped with the software TOCA (S. Zaefferer) for on-line crystallographic analysis. Furthermore, an X-ray goniometer equipped with capillary beam guide for high X-ray intensity and an area detector is available. Alternatively, this instrument can be used with a monochromator set-up for high-precision residual stress measurements.

**Highlights 2007-2008.** A central project during the past 2 years was the optimization and application of the newly developed 3D EBSD-based orientation microscopy system which allows the 3-dimensional analysis of microstructures and crystallographic microtextures, Fig. 5 [2,3,21,22,30]. The technique is based on the fully automated integration of serial sectioning using a focused ion beam and characterisation of the individual sections by EBSD. It is realized in a Zeiss Crossbeam XB1560 FIB (combination of a high resolution SEM and a focussed ion beam (FIB)). By this method, spatial resolutions of about  $50^3 \text{ nm}^3$  are achieved and material volumes of up to about  $100 \times 30 \times 30 \text{ }\mu\text{m}^3$  can be probed.

The technique was successfully used to observe recrystallisation nucleation mechanisms in a heavily cold rolled Fe 36 % Ni alloy that had been annealed with different soaking times. The 3D EBSD measurements were subsequently used as input into a Monte-Carlo-based Potts model simulation of subgrain growth which allows to follow the evolution of the grain topology, texture, and stored energy in the material. More details on this project can be found in part III of this volume.

Another highlight during the past two years concerns the improvement of the spatial resolution of the EBSD technique using a low acceleration voltage. The first promising results indicate a significant reduction of the electron-matter interaction volume already at 10 kV. We currently design a new detector and check the results at still lower electron energies.

Finally, an important project with considerable relevance also for practical steel research has been our large-area EBSD investigation which has led to

a very accurate description of the texture of coarse-grained, secondary recrystallized Fe-Si sheets with Goss texture ( $\{110\}\langle 001 \rangle$  preferred orientation). Using our newly developed technique we were able to detect very subtle differences in the texture of differently treated FeSi material and relate them to the final magnetic properties of the sheet.

### Thermomechanical Treatment (D. Ponge)

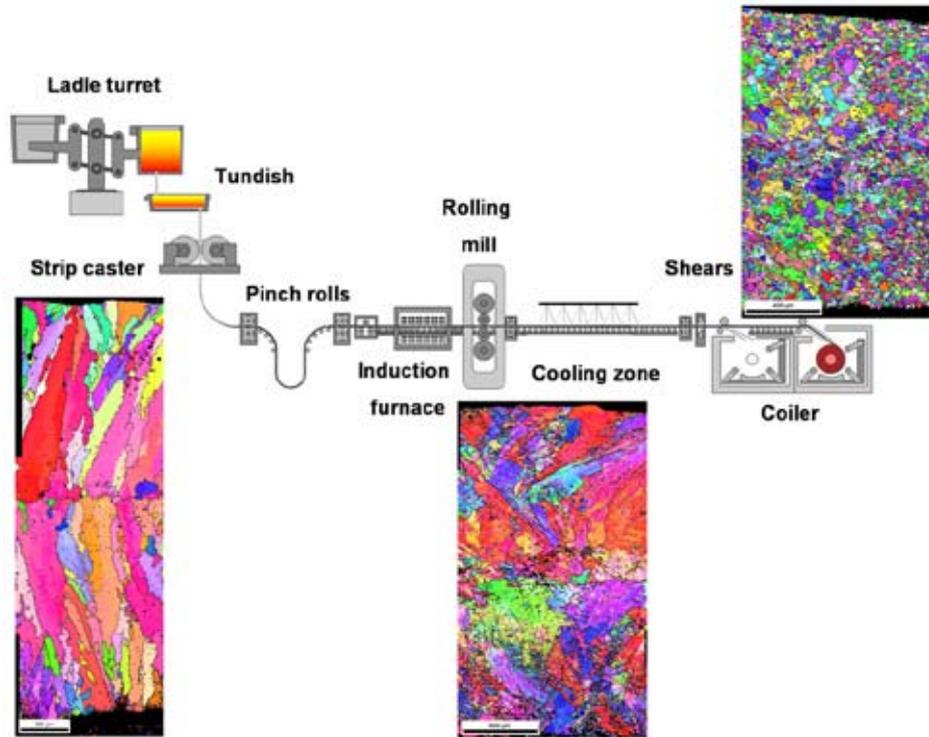
#### Group Mission and Interdisciplinary Activities.

The group for Thermomechanical Processing conducts basic studies on the relationship between processing, microstructure, and mechanical properties of steels. The characterization and understanding of the mechanisms that create certain microstructures under complex thermomechanical history and boundary conditions and their relevance for the observed micro- and macromechanical behaviour are key areas of the work.

Particularly advanced high strength alloys such as TWIP, TRIP, stainless, and multiphase steels receive their properties from a delicate interplay of a complex non-equilibrium thermodynamic constitution (different phases and their local composition) on the one hand and a number of deformation and respectively transformation mechanisms (e.g. twinning, shear band formation, dislocations, grain boundaries, phase boundaries, precipitates, effects of interstitials, martensite formation, upper and lower bainite to name but a few essential ones) on the other hand.

In order to invoke certain deformation mechanisms for achieving a desired mechanical behaviour in a new alloy a certain well-designed kinetic path must be developed and rendered into a thermomechanical process (imposing joint mechanical and thermal boundary conditions) for creating the desired phases and local chemical composition. These different phases are the carriers of certain specific deformation mechanisms which finally lead to a desired mechanical behaviour. An example is the thermomechanical processing of a TRIP steel which, under load, produces nano-twins, twin bundles, stacking faults, and  $\epsilon$ -martensite which - when intersecting - lead to the formation of  $\alpha'$ -martensite nuclei. The transformations in steel processing are usually non-equilibrium transformations and the systems encountered are multi-component alloys so that the design of a particular thermomechanical process for a desired property spectrum plays a key role in steel design.

Experiments are conducted by using a variety of casting and large scale forming devices, such as the 2.5 MN hot compression machine. This servohydraulic press is capable of conducting



**Fig. 6:** EBSD maps revealing the inheritance of microstructure and crystallographic texture in the course of a strip casting and subsequent in-line hot rolling and coiling process of an austenitic stainless steel [38].

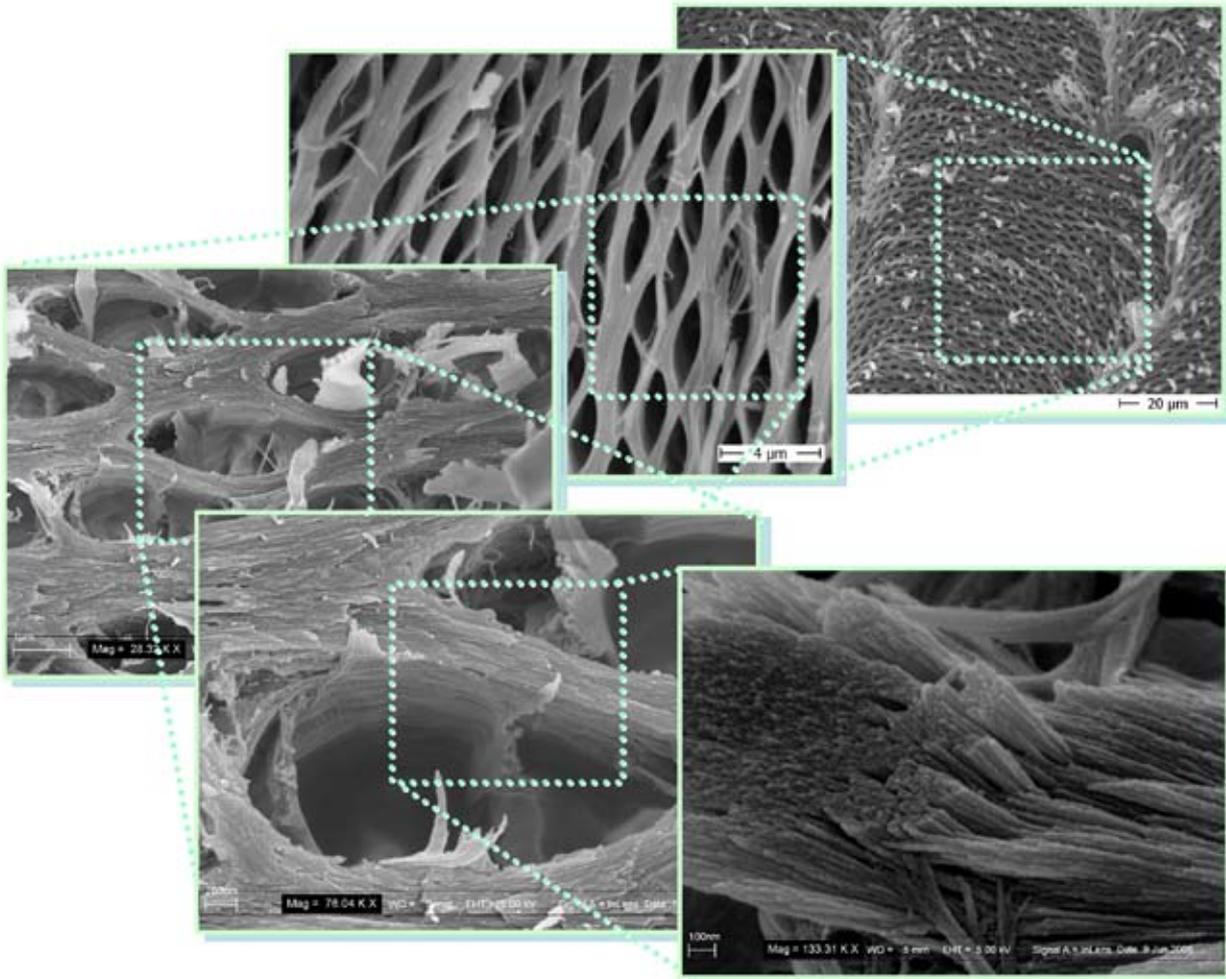
large-scale thermomechanical processes by performing multi-step hot flat compression tests as a realistic approximation of industry-scale hot forming operations. Characterization is conducted in close cooperation with the other groups by applying the full spectrum of experimental techniques available in the institute including deformation dilatometry, electron microscopy, and mechanical testing.

**Highlights 2007-2008.** The key projects in the past two years were concerned with ultra fine grained steels [31] and stainless steels. Microstructures comprising fine ferrite grains and dispersed cementite particles for plain C-Mn steels (with different contents of C and Mn) have been achieved by controlled cooling and heavy warm compression on a 2.5MN hot compression set-up. The final re-coiling has also been investigated as an important step for large scale production. The grain boundary character distribution created in such steels was investigated by using electron backscattering diffraction (EBSD): The fraction of high angle grain boundaries (disorientation angle  $\omega \geq 15^\circ$ ) was found to be approximately 60% in the ferrite. Considering only high angle grain boundaries the average ferrite grain size amounted to only 900 nm. If all grain boundaries ( $2^\circ \leq \omega \leq 63^\circ$ ) are considered, the mean ferrite (sub)grain size was only 600 nm. Dispersed fine cementite particles were found to be very efficient in preventing ferrite growth. A second set of projects investigated novel process routes which entailed ultra fine microstructures which were formed by thin slab casting combined

with direct rolling strategies in the austenitic regime. Another project is concerned with applying such ultra-fine grain strategies also to dual phase steels [32,33]. In the field of ultra high strength steels the beneficial effect of thermomechanical treatments on the mechanical properties of spring steels was demonstrated and different treatments were designed [34-37]. These treatments enable lower tempering temperatures for these martensitic steels resulting in higher strength with tensile strength more than 2000 MPa. At the same time the ductility is improved and the susceptibility to embrittlement by harmful elements like phosphorus, copper and tin is reduced. Regarding the activities on stainless steels attention was mainly placed on detailed studies of the inheritance of microstructure and texture in the course of casting and subsequent hot rolling strategies, Fig. 6.

**Biological Materials (S. Hild)**

**Group Mission and Interdisciplinary Activities.** The objective of the group is to study the relationship between the hierarchical internal structure and the micromechanical behaviour of natural materials [39-42]. The emphasis is on chitin-based materials such as in the exoskeleton of arthropods and, to a minor extent, collagen-based materials such as bone. Particular attention is placed on the investigation of the structure and the mechanical properties of crustaceans, mainly of *homarus americanus* (lobster). This material is an excellent example of a bio-nano-



**Fig. 7:** Sequence of micrographs from the microscale down to the nanoscale that reveal some features of the hierarchical structure of the cuticle of lobster. More details of the underlying theoretical treatment of the composite mechanical response and also on the structure details are presented in the form of a brief review article on p. 169. The theoretical analysis of the structure-property relations and the investigation of the basic electronic structure of the chitin crystals is conducted in close cooperation with the Department for Computational Materials Design (Prof. Neugebauer).

composite with excellent mechanical properties. As in most mineralized natural polymer tissues, the lobster cuticle has a complex hierarchical organization and consists of the structural biopolymer chitin in the form of reinforcing crystalline nanofibrils, various proteins, mineral nanoparticles (mostly amorphous calcium carbonate (ACC) but also some crystalline calcite), and water, Fig. 7 [43-47].

A better understanding of the structure/property relations in such mineralized biological tissues at different length scales not only brings an insight about how nature designs structural materials with specific functions but it can also inspire the bio-oriented design of a next generation of advanced materials for applications in nanotechnology, structural and medical tissue engineering, and armour design. In that context the cuticle of the lobster *Homarus americanus* can serve as a model material because of the availability of extensive experimental data about its microstructure, crystallographic texture and mechanical properties recently obtained by the group.

**Highlights 2007-2008.** In the past two years the group has pursued two main directions, namely, the experimental characterization of the lobster cuticle and the modelling of the relationship between its complex hierarchical structure and the resulting mechanical properties [39-42].

The experimental investigations have concentrated on the structure and the mechanics of the lobster claw and carapace. For the structure and microstructure characterization experiments we used light optical transmission microscopy, laboratory-scale X-ray diffraction in conjunction with an area detector, synchrotron-based phase and texture analysis at DESY and DELTA, high resolution scanning electron microscopy, and transmission electron microscopy.

For deriving the multiscale structure-property relations of the cuticle, we developed together with the department for Computational Materials Design a hierarchical homogenization method including ab initio calculations without free parameters at the



molecular level and a mean-field homogenization approach for higher hierarchy levels. The elastic constants of crystalline  $\alpha$ -chitin – a structural polysaccharide polymer similar to cellulose and found in all arthropod cuticles – were calculated ab initio for the first time. The fundamental level in our multi-scale and multi-disciplinary analysis lies at the nanoscale where the electronic structure and inter-atomic bonding of the studied material determine the elastic properties. We used a hierarchical approach that combines Valence Force Field Molecular Dynamics calculations with Tight Binding and Density functional Theory calculations in order to determine the complete set of lattice- and elastic constants of single-crystalline  $\alpha$ -chitin with orthorhombic unit cell. These results are then used as input in continuum-based homogenization schemes that are based on the Mori-Tanaka and Torquato 3-point approaches. Details of the work are presented in form of a short highlight article on p. 169.

### Max-Planck-Fraunhofer Group on Computational Mechanics of Polycrystals (P. Eisenlohr)

#### Group Mission and Interdisciplinary Activities.

The group for Computational Mechanics of Polycrystals was established in 2005 as the first ever joint research group between the Max-Planck-Society (Department of Microstructure Physics and Metal Forming, MPIE) and the Fraunhofer-Society (Fraunhofer-Institut für Werkstoffmechanik IWM, Freiburg). It develops theoretical approaches for the mechanics and damage initiation of polycrystalline matter with the aim to promote its use for industrial applications such as encountered in the fields of aerospace, automotive, and medical engineering.

Most metals used in structure applications are polycrystals, i.e., they contain a very large number of grains. The elastic-plastic response of an individual grain is anisotropic due to its crystalline structure. Such anisotropy is noticeable for polycrystals in which the crystallite orientation distribution (crystallographic texture) of the material is not random. It should be noted that practically all process routes lead to texture evolution. Predicting the texture evolution during forming of metallic materials is of high economical interest. An example is sheet metals produced by rolling which typically possess strong textures. The resulting anisotropy influences the ensuing deformation, with consequences for tool design, crash behaviour or surface quality of final products such as for instance automotive body parts.

Modelling the interaction of lattice slip and evolving grain orientations in a polycrystalline setting is a strong challenge. The reason is that in a polycrystal the neighbourhood around individual grains as well as their shape varies from grain to grain. Therefore, the local boundary conditions experienced by each grain

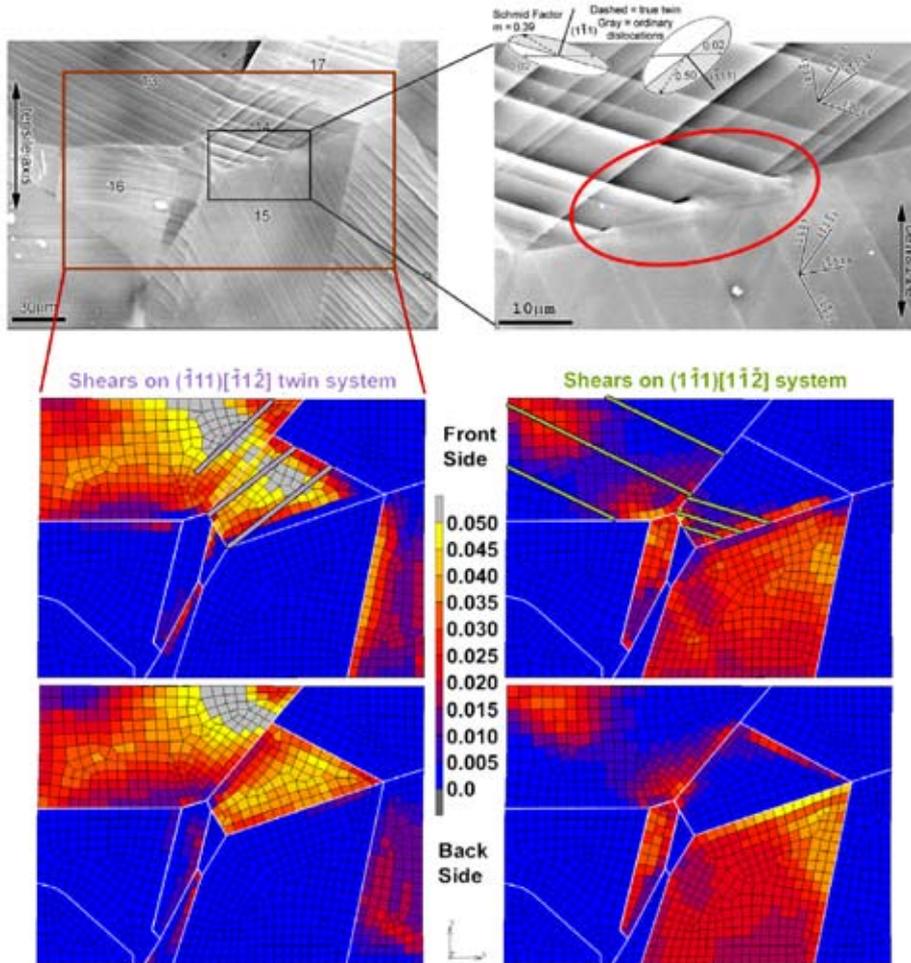
differ more or less strongly from the global average boundary conditions applied to the polycrystal as a whole. Solving this boundary value problem with all degrees of freedom present in a polycrystal is certainly unfeasible when dealing with several million grains. Hence, simplifications are required to be able to model and predict the coupled deformation behaviour and texture evolution at a continuum material point.

In this context a number of homogenization schemes were developed which make certain assumptions on the interactions among neighbouring crystals under a global loading state. Typical examples are the relaxed-constraints Taylor models which drop the strict Taylor equal strain assumption among the grains (ensuring compatibility of the deformation) and allows for local misfit strains among the crystals. In such classical homogenization models only global relaxations are considered, but true grain interactions are neglected. Grain cluster models represent a versatile class of approaches. They compose a mesoscopic aggregate of interacting grains (typically two or eight crystals) for which the boundary conditions are jointly imposed. The deformation can be relaxed among the grains within a cluster as long as the external boundary conditions are fulfilled for the entire cluster [48].

In this scientific setting the mission of the group lies in developing novel homogenization schemes for polycrystal mechanics with respect to applications in advanced engineering applications. In terms of metal physics the group places its current focus on the micromechanical fundamentals associated with multiphase mechanics, deformation induced phase transformations, deformation twinning, and strain-induced damage nucleation.

**Highlights 2007-2008.** In the past 2 years the group has pursued mainly three aims. The first one was the development of an advanced approach to the deformation simulation of clusters of crystals which is referred to as Relaxed Grain Cluster model (RGC). This approach does not only improve existing homogenization schemes for polycrystal mechanics but it can also be used as a homogenization method for multiphase polycrystalline materials. In part III of this report more details on our studies on cluster mechanics are presented in form of a short overview article (see p. 221).

The second aim of the group was the development of advanced constitutive models that describe the individual deformation behaviour inside the grains. This work is closely linked to the activities of the group Theory and Simulation (F. Roters). Of particular interest was the development of models describing deformation via twinning and martensite formation and its interaction with dislocation slip. All activities were closely connected via frequent



**Fig. 8:** Upper row: Electron Channing Contrast Image (ECCI) of microcracks formed in TiAl between grains 14 and 15, while boundaries between grains 13 and 16, and between 14 and 17 (an annealing twin) remained intact after a surface strain of about 1.4%. The microstructure within the larger box in the upper figure was modelled with a crystal plasticity finite element mesh (CPFEM). The 4 bottom images show the shear contributions on two main slip systems on the front and back sides. Three of the microcracks can be correlated with twin intersections [49,50].

meetings with the group at the Fraunhofer-Institut für Werkstoffmechanik. The group has recently successfully passed the second review of the two funding agencies (MPG, FhG) and received further support until 2011 (see also p. 29).

The third recent direction of the group was to elucidate the role of heterogeneous deformation on damage nucleation at grain boundaries in single phase metals. This work is done in close cooperation between our department (Dr. Eisenlohr) and Professors T.R. Bieler and M.A. Crimp from Michigan State University in East Lansing, USA, and Professor D.E. Mason from Albion College in Albion, USA. The project aims to better understand where and why cracks or voids form in a loaded polycrystal particularly at grain or phase boundaries. Interfaces are often invoked as the reasons for critical damage nucleation, but so far without any quantification or detailed mechanical understanding. In the project, which is jointly funded by the US and German research foundations (NSF, DFG, Materials World Network programme, see p. 27), we investigate

deformation at or near grain boundaries, including slip transfer and heterogeneous deformation, using both, electron microscopy and crystal plasticity-finite element simulations, Fig. 8 [49,50].

### MMM-Group on the Theory and Simulation of Complex Fluids (F. Varnik)

**Group Mission and Interdisciplinary Activities.** Some condensed phases of matter are neither simple liquids nor simple crystalline solids. Rather, mechanical properties of these complex fluids are intermediate between ordinary liquids and ordinary solids. In many cases, complex fluids are easily deformed under the action of rather weak forces [51]. As an example, it is often sufficient to shake a colloidal solid in order to make it flow (shear melting). In many cases, shear melting is accompanied by a decrease of shear viscosity upon increasing shear rate (shear thinning). More generally, the relation between the stress and deformation for a complex fluid is often non-linear [51]. The practical importance of shear melting and shear thinning phenomena can



hardly be overemphasized, since, without these effects, it would be nearly impossible to process many of the soft materials used in industry. One of the research topics of the group is the relation between the microscopic dynamics (e.g. dynamics of structural relaxation) and the rheological response of complex fluids such as polymers [52-54] and colloidal dispersions via the study of relatively simple computer model systems [55,56]. The aim of these studies is to isolate generic features from system specific one and to examine predictions of microscopic theories [57-59] in this fast evolving research field [60,61]. A particularly interesting aspect is the interplay between the dynamics of structural relaxation and the response to an external perturbation.

Using the lattice Boltzmann method and variants thereof, the group also investigates issues relevant in microfluidics such as inhomogeneous diffusive broadening [62], droplet dynamics on chemically heterogeneous substrates [63] as well as flow between topographically rough walls [64-65]. One of the basic questions is how related driving/pinning forces (which may drastically modify the droplet dynamics) depend on the geometry of the channel and the liquid-substrate wetting properties. The aim is to use the obtained knowledge in order to optimize the system parameters (e.g. minimize dissipation losses and improve the effect of chemical gradients, if the latter is used to guide the flow).

The group on Complex Fluids has been established as part of the Max-Planck-Multiscale Modelling Initiative for a period of 4 years, 2005-2009. It is linked both to the other theory activities in our department and to the group of M. Rohwerder in the Department for Interface Chemistry and Surface Engineering (Prof. Stratmann) concerning studies of coating and wetting phenomena via a combined theory/experiment approach. Starting from Oct. 2008, the group leader, Dr. habil. F. Varnik has accepted a group leader position at the ICAMS centre at Ruhr-Universität Bochum (ICAMS: Interdisciplinary Centre for Advanced Materials Simulations, see p. 17) so that the future work of this initiative will take place at both locations (Bochum, Düsseldorf).

**Highlights 2007-2008.** During the past 2 years the group was mainly concerned with two aims. The first one was the development of advanced lattice Boltzmann methods and the second one was the work on the physics of complex fluids.

As to the first goal, efficient parallel lattice Boltzmann tools have been developed for the study of a number of issues such as inhomogeneous diffusive broadening [62], dynamics of droplets on chemically patterned substrates [63] as well as roughness-induced flow instability [64,65].

Even in the simplest case of a laminar flow in a rectangular channel, our studies of broadening of a passive tracer field entering the channel via, say, the left half of the channel's cross section uncover the existence of different regimes for the dependence of the width of the inter-diffusion zone on the distance from the entrance of the channel. While tracers follow the normal diffusive law along the channel's axis (away from the walls), a sub-diffusive regime is observed for tracers close to the walls [62]. This result is of considerable importance in cases where one is interested in an understanding of the distribution of tracer field close to the channel walls such as in catalytic reactions and surface patterning.

Our studies of droplet dynamics on chemically patterned substrates, on the other hand, reveal qualitatively different behaviour for the case of slit geometry as compared to open substrates. As shown in our recent work [63], the use of a slit geometry significantly enhances the surface effects on droplet dynamics. Importantly, this effect can not simply be traced back to the factor of 2 in the number of substrates. Rather, it is the change in the droplet shape that lies at the heart of this difference. In addition to confirming our estimates obtained via simple scaling arguments, we show how well designed experiments in conjunction with mesoscopic computer simulations may shed light onto many fundamental aspects of fluid dynamics on chemically patterned substrates. In particular, the maturity of multi-phase lattice Boltzmann method in tackling the correct dynamic behaviour on substrates with spatially variable wettability is demonstrated in this article [63].

Another interesting phenomenon observed within our lattice Boltzmann simulation is the so-called roughness-induced flow instability [64,65]. It is important to note that similar effects have later been observed in recent experiments performed by Dr. Tamas Harasti (group of Prof. Spatz) at the University of Heidelberg thus emphasizing the predictive power of the lattice Boltzmann method further. We are at the moment working on a further development based on a consequence of this very interesting effect, namely the possibility to prevent flow instability (and hence reduce dissipation loss) via a suitable design of the channel walls.

An elegant way to explore the properties of complex fluids is to subject them to an external driving force (such as a simple shear) and survey their response. In the unperturbed state, the material can assume, depending on temperature, different states ranging from the supercooled (liquid-like) state to the glassy (solid-like) phase. This has strong implications for the response of the system to an external drive such as a spatially homogeneous shear. In the supercooled state, the viscosity shows a shear



thinning behaviour for shear rates higher than the inverse of some characteristic relaxation time but then converges towards a constant value (linear response) as the shear rate is reduced below this value. In contrast to this, in the glassy phase, the shear viscosity ideally diverges as  $1/\text{shear rate}$  in the limit of vanishing shear rate. Thus, the absence of a constant viscosity regime can be considered as a hall mark of the glassy state [60]. Very recently, we made a significant contribution to a better understanding of

the microscopic origin of this very interesting behaviour by showing that a simple schematic model based on the competition between the following two opposing effects is sufficient for an excellent description of the rheological response of the system for the entire range of temperatures and shear rates studied: Freezing of the so-called nearest neighbour cage upon cooling on the one hand and the gradual destruction of this cage via shear advection of density fluctuations on the other hand [61].

## Spirit, Outreach, Achievements

Projects within our department and also among the departments are pursued in an interdisciplinary and team-oriented spirit. Scientists in our department come from such different backgrounds as physics, materials science, metallurgy, biology, informatics, chemistry, and mechanics. Projects are conducted in an atmosphere of mutual inspiration, discussion, and cooperation. Paramount to the success of our work is the close exchange among theorists and experimentalists.

The working atmosphere in our group was during the past 2 years dominated by an international flair bringing together young scientists and visiting scholars from Algeria, Australia, Belgium, Brazil, Bulgaria, China, Egypt, France, Germany, India, Indonesia, Israel, Iran, Japan, Jordan, Korea, Nigeria, Russia, Sweden, Spain, Thailand, Turkey, UK, USA, and Venezuela, Fig. 9. Our international orientation is also reflected by our extramural cooperation partners, namely, Prof. Bleck and Prof. Gottstein (RWTH Aachen, Germany), Prof. Rollett (Carnegie Mellon University, USA), Prof. Radovitzky (MIT, USA), Prof. Mao (University of Science and Technology Beijing, China), Prof. Sandim (University of Lorena, Brazil), Prof. Bieler and Prof. Crimp (Michigan State University, USA), Prof. Mason (Albion College, USA), Prof. Hono and Prof. Adachi (National Institute for Materials Science, Japan).

The international visibility of the department is reflected by more than 15 plenary and keynote lectures at international conferences in the past 2 years, numerous memberships of scientists from the department in international editorial, conference, and advisory boards, and a number of awards with which the department was honoured in the last 2 years. For instance M. Calcagnotto was awarded with a grant from the Japan Society for Promotion of Science (JSPC); F. Varnik received the Best Talk Award at the Nano- and Microfluidics Conference in Bad Honnef; F. Weber provided the figure for

the front cover of *Advanced Engineering Materials* Vol. 10, No. 8, August 2008; L. Raue received the Outstanding Student Researcher Award at the 15th International Conference on Textures of Materials (ICOTOM 15) in Pittsburgh, USA; C. Zambaldi was awarded with the 1st prize in the Poster Competition of the RMS Electron Backscatter Diffraction Meeting in Sheffield, UK; T. Gronau was honored by the Max-Planck-Society with the Outstanding Apprentice Award; D. Raabe was elected as a member of the Nordrhein-Westfälische Academy of Sciences; D. Raabe received the Lee Hsun Lecture Award of the Institute of Metal Research of the Chinese Academy of Sciences in Shenyang, China; K. Verbeken and D. Raabe received the Best-Poster-Award for their contribution "Identification of  $\epsilon$  martensite in Fe-based shape memory alloys by means of EBSD" at the E-MRS 2007 conference in Warsaw, Poland; D. Raabe was elected as a Member of the Board of Governors of Acta Mater. Inc., USA; D. Raabe was elected as a member into the Selection Committee of the Alexander von Humboldt Foundation, Germany; C. Zambaldi, F. Roters, T. R. Bieler and D. Raabe were awarded by the 11th World Conference on Titanium (JIMIC5) with the Best Poster Award in Kyoto, Japan; C. Zambaldi was awarded with a grant research fellowship from the Japan Society for Promotion of Science (JSPC) to the "National Institute for Materials Science (NIMS)"; M. Frommert, D. Dorner, L. Lahn, D. Raabe, and S. Zaefferer received the "Best Poster Award" for their contribution "3D Investigation of Early Stages of Recrystallization in Deformed Goss-Oriented Fe<sub>3</sub>Si Single Crystals" at the "Third International conference on Recrystallization and Grain Growth, ReX & GG III in Korea; P. Eisenlohr was elected as an AICES "Young Researcher"; P. Romano was awarded a grant research residency from the Japan Society for Promotion of Science (JSPC) to the "National Institute for Materials Science (NIMS)"; C. Sachs was awarded with a Max-Planck Minerva Short-Term Research Grant; C. Sachs was



Fig. 9: *The team.*

awarded with the best paper award by TMS for the paper “Homogeneous Steel Infiltration”; B. Stieger was honored by the Max-Planck-Society with the Outstanding Apprentice Award; A. Brahme received the Annual Student Award from Acta Mater. for his contribution “3D reconstruction of microstructure in a commercial purity aluminum” Scripta Materialia 55, 2006, 75-80, September 2007 at the MS&T Conference, Detroit, USA; and H. Faul received the honorary needle in silver for his 10 years of commitment in professional training by the Industrie- und Handelskammer, Stuttgart. All these honours were received during the years 2007 and 2008.

Besides these awards two habilitation procedures out of the Department for Microstructure Physics and Metal Forming were initiated during the past 2 years (F. Varnik, S. Zaefferer).

A number of senior scientists received professorship positions during the past 2 years: Dr. Y. Cao became a Professor for Soft Matter Mechanics at Tsinghua University in China; Dr. S. Hild became Professor for Polymer Science at Linz University in Austria; Dr. N. Zaafarani became Professor for Materials Mechanics at Tanta University in Cairo; Dr. S. Nikolov will become Professor at the Bulgarian Academy of Science in Sofia; and Dr. Z. Huang became Professor for Metallurgical Modelling at Shanghai Jiao Tong University in China.

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## Research Projects in Progress

### Theory and Simulation

*Demir, Roters Raabe:* Advanced constitutive laws based on geometrically necessary dislocations for small scale crystal plasticity finite element simulations

*Demir, Roters Raabe:* Micro- and nanobeam compression, experiments and simulations

*Demir, Zaafarani, Roters, Raabe:* 3D experiments and simulations on nanoindentation

*Dmitrieva, Dondl\*, Müller\* (\*MPI Leipzig), Raabe, Roters:* Experiments and modelling of deformation-induced patterning (lamination) phenomena in single crystal shear deformation

*Friák, Ma, Sander, Neugebauer, Raabe:* Combination of ab-initio simulations of lattice parameters, elastic properties, and phase stability and crystal plasticity finite element simulations for Ti-base alloys

*Lymperakis, von Pezold, Neugebauer, Raabe:* Molecular dynamics simulation of grain boundary motion

*Ma, Demir, van Swygenhoven\*, Maas\* (\*Paul-Scherer Institut, Switzerland), Raabe, Roters:* Crystal plasticity finite element simulation of the boundary conditions and orientation dependence of pillar compression

*Ma, Friák, Neugebauer, Raabe:* Using ab-initio simulations for the simulation of parameters for solid solution hardening

*Petrov, Friak, Lymperakis, Nikolov Neugebauer, Sachs, Fabritius, Raabe:* Combination of ab-initio simulations and continuum models in mineralized chitin-based biological materials

*Roters:* Development of texture-function-based crystal plasticity finite element models

*Schulz, Winning, Roters, Raabe:* Cellular automata for recrystallization simulation

*Steinmetz, Roters, Raabe:* Constitutive modelling of TRIP and TWIP steels based on dislocation theory

*Tikhovskiy, Roters, Raabe:* Crystal plasticity finite element simulations of deep drawing of stainless steels

*Weber, Raabe:* Simulation of nanobeam bending

*Winning, Raabe:* Fast algorithms for physically-based recrystallization simulations in metals

*Winning, Raabe:* Recrystallization simulation under consideration of the mobility of low angle grain boundaries

*Zaefferer, Thomas:* Monte Carlo Potts modelling for recrystallization nucleation and subgrain growth simulations

*Zambaldi, Roters:* Crystal plasticity finite element simulations of plastic deformation of TiAl

*Zhao\*, Radovitzky\* (\*MIT, USA), Roters, Raabe:* Massively parallel 3D crystal plasticity finite element simulations

### Diffraction and Microscopy

*Balasundaraman, Cao, Nikolov, Raabe:* Nano-indentation and nano-scratching in thin polymer layers

*Bastos, Zaefferer, Ponge, Raabe:* Microtexture and microstructure analysis in multiphase steels

*Bastos, Zaefferer, Raabe:* 3D texture and microstructure analysis of electrodeposit Nickel-Cobalt nanocrystals

*Frommert, Zaefferer, Günther\* (\*TKES, Germany), Lahn (TKES, Germany), Raabe:* Texture and microstructure evolution in silicon steels

*Imlau\*, Bleck\* (\*RWTH Aachen, Germany), Zaefferer:* Texture and microstructure of high-strength multiphase steels

*Khorashadzadeh, Winning, Zaefferer, Raabe:* 3D EBSD analysis of Cu-alloy processed by ECAP

*Ma, Sander, Raabe:* Microstructure, phase stability, and elastic properties of beta-Titanium alloys

*Perranio, Roters, Raabe:* Texture and microstructure evolution in dual phase steels

*Romano, Friedel\* (\*TKS, Germany), Zaefferer, Raabe:* Microstructure of high-strength multiphase steels

*Sandim\* (\*Univ. de Lorena), Raabe:* Microtexture in Niobium

*Sato, Zaefferer:* Investigation of orientation relationships in diffusional and martensitic phase transformations

*Song, Raabe, Zaefferer, Kostka:* Microstructure and texture at explosive cladding joint welds

*Steinmetz, Zaefferer, Raabe:* High resolution EBSD

*Tao, Mao\* (\*TU Beijing), Raabe:* Texture of diamond thin films

*Weber, Raabe:* Nanobeam bending experiments

*Yi, Zaefferer:* Microscopic deformation mechanisms in Magnesium

*Zaefferer, Demir, Zaafarani, Bastos, Raabe:* 3D EBSD analysis by joint focused ion beam and electron back scatter diffraction microscopy

*Zaefferer, Kobayashi:* Phase analysis for multiphase steels by use of a multiphase EBSD method applied to diffusion couple samples

### Thermomechanical Treatment

*Calcagnotto Ponge, Raabe, Adachi\* (\*NIMS, Japan):* Microstructure and properties in ultra fine grained dual phase steels

*Herrera, Ponge, Raabe:* Texture evolution in dual-phase stainless steels

*Lei, Barani\* (\*TKN Bochum), Ponge, Raabe:* Texture evolution in ferritic stainless steels

*Ponge, Raabe:* Processing and microstructure of highly alloyed ferritic and austenitic stainless steels

*Ponge, Raabe:* Processing and microstructure of thermomechanically deformed TWIP steels

### Biological Materials

*Fabritius, Sachs, Hild, Raabe:* Structure and micro-mechanical properties of the lobster cuticle.

*Nikolov, Fabritius, Raabe:* Constitutive micro-mechanical modelling of bone mechanics.

*Raue, Al-Sawalmih\*, Fratzl\*, Paris\* (\*MPI Golm), Fabritius, Raabe:* Crystallographic texture of the cuticle of lobster

*Sachs, Fabritius, Raabe:* Micromechanical properties of bone

*Swadener\* (\*Los Alamos, USA), Raabe:* Mechanics of lipid membranes

*Wu, Fabritius, Raabe:* Microstructure of biological photonic crystals

### MPG-FhG-Group on Computational Mechanics of Polycrystals

*Eisenlohr, Bieler, Crimp\* (\*Michigan State Univ., USA), Mason\* (\*Albion College, USA), Roters, Raabe:* Strain-induced damage nucleation in experiment and crystal plasticity finite element constitutive models

*Eisenlohr, Liu, Lebensohn\* (\*Los Alamos, USA), Roters, Raabe:* Polyphase and polycrystal homogenization theory based on fast Fourier approximations and its incorporation into finite element simulations

*Hantcherli, Eisenlohr, Roters, Raabe:* Consideration of deformation-induced twinning in crystal plasticity finite element constitutive models

*Tjahjanto, Eisenlohr, Roters, Raabe:* Polyphase and polycrystal homogenization theory based on grain cluster relaxation models

### MMM-Group on the Theory and Simulation of Complex Fluids

*Ayodele, Varnik, Raabe:* Transverse diffusive broadening in pressure driven microchannels: A lattice Boltzmann approach.

*Varnik, Raabe:* Boltzmann lattice gas models for blood flow simulations

*Varnik, Raabe:* Simulation of scaling effects in nano- and microscale fluid dynamics at deformable metal surfaces

*Varnik, Truman, Wu, Uhlmann, Rohwerder, Raabe, Stamm:* Boltzmann lattice gas models of wetting phenomena

### Others

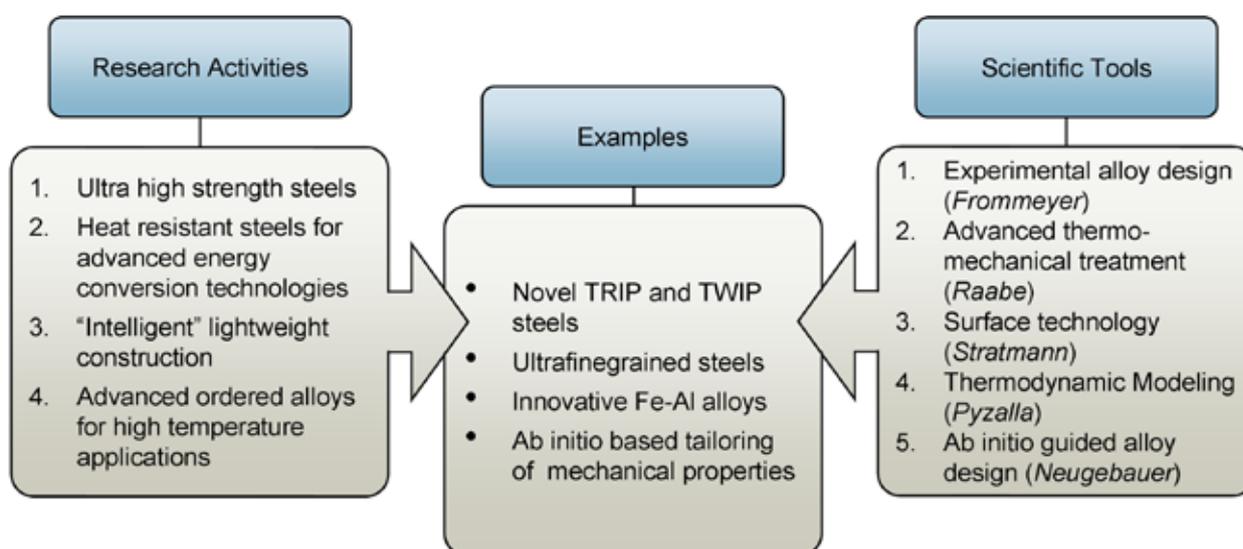
*Hono\* (\*NIMS, Japan), Raabe:* Microstructure of wire drawn ternary high strength and high conducting Cu-base nanocomposites



## PART III.

# INTER-DEPARTMENTAL RESEARCH ACTIVITIES – SELECTED HIGHLIGHTS

## New Structural Materials



### Selected Highlights 2007/2008

Development and Characterization of High-Strength Mn-Al-C Light-Weight TRIPLEX Steels <i>R. Rablbauer, U. Br�ux, G Frommeyer</i>	123
Design of Heat-Resistant Steels Supported by Thermodynamic and Kinetic Modelling <i>J. Garcia, D. Rojas, O. Prat, R. Maccio, A.R. Pyzalla</i>	127
Combining Experimental and Computational Methods in the Development of Fe <sub>3</sub> Al-based Materials <i>J. Deges, M. Fri�k, G. Frommeyer, J. Neugebauer</i>	131
Processing and Mechanical Properties of Ultrafine Grained Dual-Phase Steels <i>M. Calcagnotto, D. Ponge, D. Raabe</i>	135
Using <i>Ab Initio</i> Calculations in Designing bcc Mg-Li Alloys for Ultra Light-Weight Applications <i>W.A. Counts, M. Fri�k, D. Raabe, J. Neugebauer</i>	139
High Temperature B2-Ordered NiAl-Based Alloys Reinforced by Refractory Metals for Advanced Energy Conversion Technologies <i>G. Frommeyer, R. Rablbauer</i>	143





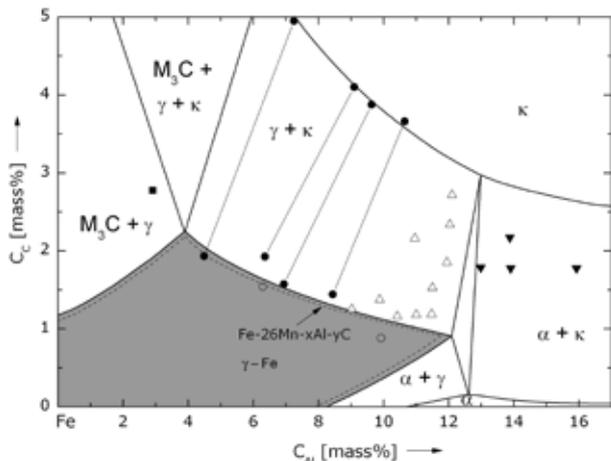
## Development and Characterization of High-Strength Mn-Al-C Light-Weight TRIPLEX Steels

R. Rablbauer\*, U. Brück\*, G Frommeyer

Department of Materials Technology

Light-weight manganese-aluminum-carbon steels of the basic compositions Fe-18/28Mn-9/12Al-0.7/1.2C possess an austenitic matrix with a dispersion of nano size  $\kappa$ -carbides and certain amounts of ferrite composing a triplex microstructure. These steels exhibit superior strength properties due to effective solid solution and precipitation hardening. The important deformation mechanism to promote enhanced ductility is homogeneous shear band formation accompanied by dislocation glide, sustained by the uniform arrangement of nano size  $\kappa$ -carbides coherent to the austenitic matrix with defined interparticle spacings.

The coexisting equilibrium phases of the manganese-aluminum-carbon steels are generally represented by the quaternary Fe-Mn-Al-C system, but a complete spatial representation is not available. Nevertheless, isothermal sections of ternary and quaternary systems, such as Fe-Mn-Al and Fe-30Mn-xAl-yC are published in literature. Fig. 1 shows the isothermal section Fe-30 Mn-xAl-yC at 900 °C of the quaternary phase diagram drawn from the literature [1]. By taking the manganese-carbon equivalents into account the  $\gamma$ -phase field is slightly contracted. The adjacent  $\gamma||\gamma$ ,  $M_3C$ ,  $\gamma||\gamma+\kappa$ , and  $\gamma||\alpha+\gamma$  equilibrium boundaries are shifting of about 0.07 mass % carbon and 0.35 mass % Al towards the center of the phase field. By taking this slightly modified

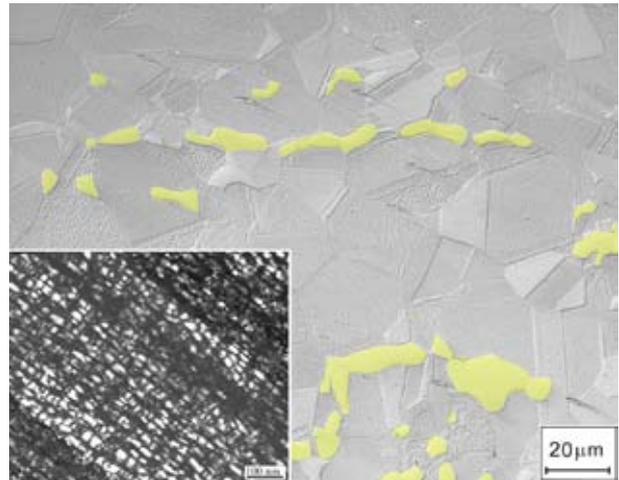


**Fig. 1:** The isothermal section of the quaternary Fe-Mn-Al-C phase diagram at 900 °C [1] illustrates the coexisting phase fields in the iron-rich corner for Fe-30Mn and varying aluminum and carbon concentrations. The phase boundaries of the  $\gamma$ -phase field of the Fe-26Mn-xAl-yC steel are marked by dashed lines.

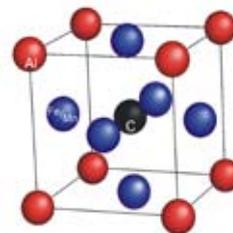
\* now at: Volkswagen AG, Konzernforschung, Wolfsburg, Germany  
\* now at: Ford Forschungszentrum Aachen, Aachen, Germany

isothermal section into account a TRIPLEX steel of the composition Fe-26Mn-10/12Al-1.2C consists of an austenitic matrix with certain amounts of ferrite and  $\kappa$ -carbides. The  $\kappa$ -carbides will be dissolved in the austenite during high temperature annealing and precipitate out by spinodal decomposition due to thermal aging at elevated temperatures of 500 to 600 °C, respectively [2].

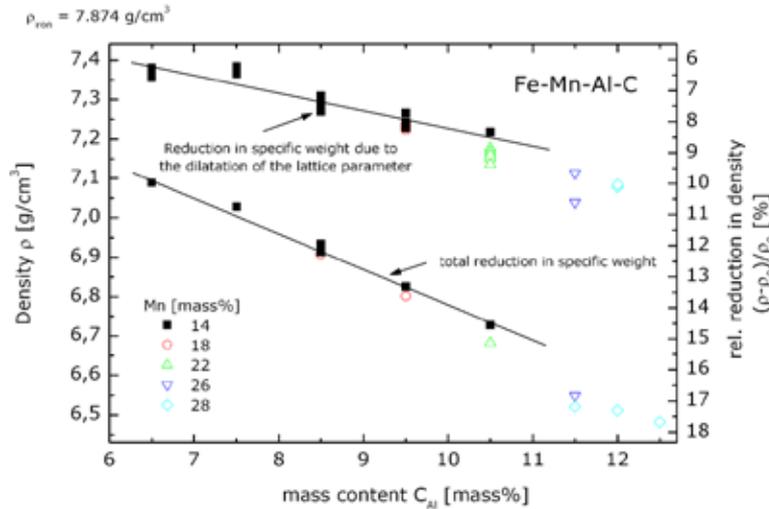
The microstructure of an as recrystallized Fe-26Mn-11Al-1.15C TRIPLEX steel sheet (Fig. 2) consists of an austenitic matrix characterized by annealing twins and about 6 to 8 vol % of ferrite. The existence of nano size  $\kappa$ -carbides within the austenite matrix is revealed by TEM dark field image inserted in Fig. 2. The  $\kappa$ -carbides precipitates are of  $L1_2$  ordered f.c.c. lattice structure as illustrated in Fig. 3.



**Fig. 2:** Optical micrograph of the microstructure of an as-cold rolled and recrystallized Fe-26Mn-11Al-1.15C TRIPLEX steel sheet. The austenite matrix reveals annealing twins and  $\alpha$ -ferrite appears as bright phase. The inserted TEM dark field (DF) image represents nano- size  $\kappa$ -carbide precipitates.



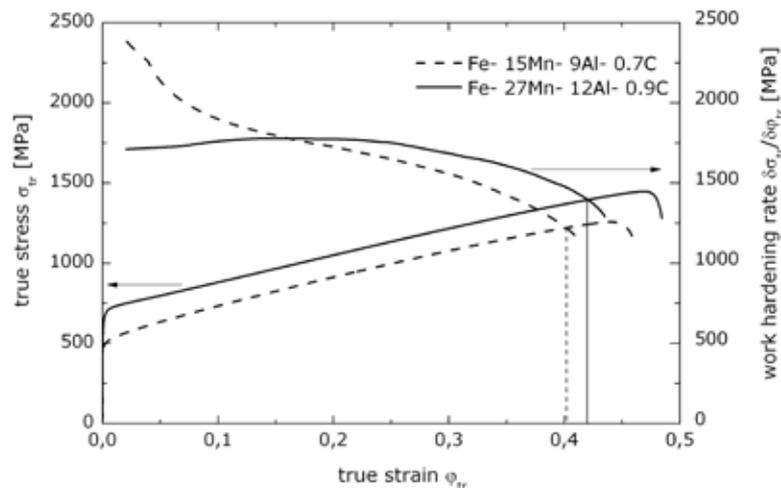
**Fig. 3:** Unit cell of  $\kappa$ -carbide  $(Fe, Mn)_3AlC$  of the ordered f.c.c.  $L1_2$  type of lattice structure. This structure is also described as  $E2_1$ -type of perovskite structure. The average lattice parameter is about  $a_0 = 0.3837$  nm and depends mainly upon the aluminium content.



**Fig. 4:** Density as function of the aluminium concentration of diverse Fe-xMn-yAl-zC steels with varying manganese contents (14 to 28 %). The lower linear curve represents the overall reduction in density with increasing aluminium concentration. The upper curve exhibits the decrease in density due to the expansion of the  $\gamma$  - lattice.

The remarkable reduction in density with increasing aluminum content of these steels is shown in Fig. 4, where the total density and the percentage relative density reduction are plotted as function of the aluminum content by taking various manganese concentrations as parameters into account. The linear relationship of the density vs. aluminum concentration is represented by the lower curve. The minimum total density amounts to  $\rho_{total} = 6.5 \text{ g/cm}^3$  at aluminum concentration of  $c_{Al} = 12 \%$ . This is equivalent to a percentage reduction in density of  $(\Delta\rho/\rho) \cdot 100 \approx 17 \%$ . The overall mass reduction of the coexisting f.c.c. and b.c.c. Fe(Mn,Al) solid solutions is due to a lower average molar mass of the alloys and a considerable decrease in the atomic density of the unit cells. This is caused by an effective lattice expansion due to the larger atomic radius of the dissolved Al atoms ( $r_{Al} = 0.147 \text{ nm}$ ) compared to the smaller iron atoms of  $r_{Fe} = 0.126 \text{ nm}$ .

The true-stress ( $\sigma_{tr}$ ) vs. true-strain ( $\phi_{tr}$ ) curves and the related work hardening rate ( $\delta\sigma_{tr} / \delta\phi_{tr}$ ) curves of room temperature tensile tests of two selected steels with the compositions Fe-15Mn-9Al-0.7C and Fe-27Mn-12Al-0.9C are depicted in Fig. 5. The Considere criterion [3] defines the transition from plastic stability to instability where the work hardening rate is equal to the true flow stress, expressed by the relationship  $d\sigma_{tr} / d\phi_{tr} = \sigma_{tr}$ . The presented linear true flow stress vs. true plastic strain curves exhibit a stress increase by a factor of 2.1 to 2.6, respectively. In order to achieve large uniform plastic elongations the strain hardening rate should be considerably high to prevent local necking in the gauge length of tensile samples. The intersections of the true stress vs. true strain curves with the related work hardening rate curves are within the range of  $0.38 \leq \phi_{tr} \leq 0.42$ . This shows that the Considere criterion is fulfilled for large plastic strains and reveals clearly the superior



**Fig. 5:** True-stress ' $\sigma_{tr}$ ' as a function of true-strain ' $\phi_{tr}$ ' and the related work hardening rate  $\delta\sigma_{tr} / \delta\phi_{tr}$  curves of two selected high-manganese-aluminum-carbon steels.



**Fig. 6:** TEM bright-field image of shear bands on  $\{111\}$  planes in the austenitic matrix.

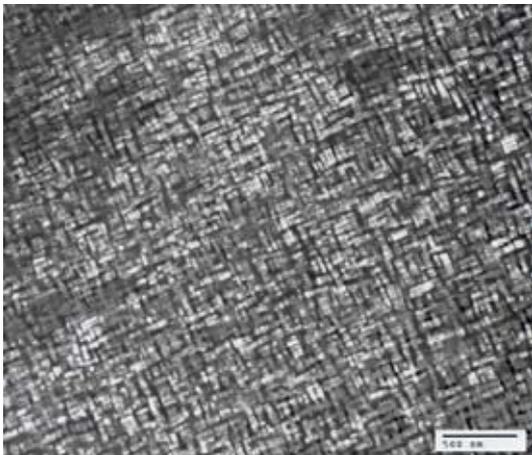
plastic potential of the high-strength TRIPLEX steels. This is similar to the supra-ductile behaviour of high-manganese TRIP and TWIP steels exhibiting multiple martensitic transformations or severe mechanical twinning [4].

At room temperature a yield strength of  $R_{p0.2} = 730$  MPa and ultimate tensile strength of  $\sigma_{UTS} = 1000$  MPa were measured. A lower differential strain hardening exponent of  $n_i = 0.58$  was derived from the stress-strain curve. This value is higher than the reported strain hardening exponents of  $0.33 \leq n \leq 0.45$  for the austenitic 304 stainless steel. TEM bright field images of tensile samples deformed at room temperature reveal in Fig. 6 uniformly arranged shear bands on  $\{111\}$ -planes of high density within the austenitic matrix. These features demonstrate the important contribution of homogeneous shear deformation to the total plastic elongation designated as **Shear Band Induced Plasticity -SIP effect-**. This is

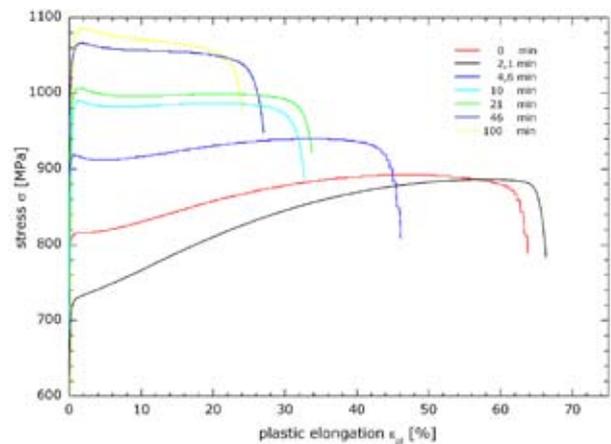
in contrast to the results of earlier investigations [2,5] where twinning or martensitic transformation were discussed as the dominant deformation mechanisms. The stacking fault energy  $\Gamma_{SF}$  of the order of  $110$  mJ/m<sup>2</sup> was estimated based on a thermodynamic model [6,7]. The  $\Gamma_{SF}$ -value indicates that the high aluminum content  $\gamma$ -solid solution matrix may not be prone to severe mechanical twinning as it is observed in genuine TWIP steels possessing much lower stacking fault energies of  $\Gamma_{SF} = 25$  to  $30$  mJ/m<sup>2</sup>, respectively [4].

Important for the formation of uniformly arranged shear bands and the strengthening of TRIPLEX steels is the regular distribution of nano size  $\kappa$ -carbides coherent to the austenite matrix, as shown in the TEM dark-field image of Fig. 7. In order to increase the strength properties and conserving sufficient plasticity the steel was subjected to thermal ageing at  $550$  °C for different time intervals between  $t_i = 2.1$  and  $100$  min, respectively. Fig. 8 represents the respective engineering stress-strain curves showing a remarkable increase of the flow stress from  $R_{p0.2} = 700$  MPa to  $1065$  MPa. It is interesting to note that the stress-strain curves of the aged samples are almost similar to those of ideal elastic-plastic solids where virtual no strain hardening occurs. This fact shows clearly the specific role of uniform shearing for achieving extended plastic elongations accompanied by moderate deformation hardening.

The promising mechanical properties of high-strength manganese-aluminum-carbon steels possessing superior ductility and reduced specific weight have attracted considerable interest of the automotive and steel industry because of several potential applications for advanced transportation systems, specifically automotive vehicles of high safety standard. An ongoing alloy development in the department of Materials Technology at MPIE is



**Fig. 7:** TEM dark field image taken using the superlattice reflection (210) of the  $\langle 001 \rangle$  f.c.c. zone axis shows the regular arrangement of  $\kappa$ -carbides of about 20-30 nm in size coherent to the austenitic matrix.



**Fig. 8:** Engineering stress-strain curves of thermally aged tensile samples of the Fe-28Mn-12Al-1C TRIPLEX steel exposed to  $550$  °C for different time intervals.



focused on the processing and characterization of microstructures, physical, and mechanical properties of these steels [8,9].

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## Design of Heat-Resistant Steels Supported by Thermodynamic and Kinetic Modelling

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Due to environmental regulations and energy saving requirements there is a strong demand for high efficiency power plant generation. Ferritic/martensitic 9-12% Cr steels are widely used for stationary steam power plants. In recent developments it was intended to rise the working temperature up to 650 °C. Increasing the working temperature reduces the long-creep and mechanical properties, especially due to coarsening of carbonitrides (i.e. MX,  $M_{23}C_6$ ) and precipitation of undesirable metastable phases (i.e. Z-phase). Due to appropriate selection of the overall composition and the thermo-mechanical treatment of the steels, the kinetic effects associated with the long-term creep at higher temperatures can be reduced. In this work the influence of the hot deformation on the mechanical properties and creep resistance of two 12% Cr heat resistant master alloys is investigated. The main objective is to produce steels with a fine dispersion of stable precipitates with small initial grain sizes, in order to increase the pinning of dislocations in the early stages of creep, or the formation of metastable phases during creep conditions (i.e. Laves phases), for strengthening of the alloy. The design is supported by thermodynamic and kinetic modelling (Thermo-Calc and Dictra) for the determination of the appropriate austenization and heat treatment temperatures, as well as for the simulation of the coarsening and phase precipitation under creep conditions.

**Experimental.** Two master alloys were prepared by vacuum induction melting with masses of about 1 kg (Tab. 1). A first set of samples was hot forged at 1150 °C (resulting in a 70% of final area reduction) with posterior air cooling. The second set was only heat treated. Heat treatments were carried out based on the information provided by the thermodynamic modelling (database TCFE5). The heat treatments

Cr	Mn	Ta	W	V	Cu	C	B	N	Si	Co
12,9	0,6	0,85	3,8	-	1	0,06	0,01	0,05	0,5	4,2
Cr	Mn	Ta	W	V	Cu	C	B	N	Si	Co
12,6	0,7	0,16	3,6	0,24	1	0,15	0,033	0,06	0,5	2,5

Tab. 1: Chemical composition of the investigated model alloys.

included an austenitisation temperature of 1070 °C for 0.5 h, subsequent air-cooling, and annealing heat treatment for 2 h at 780 °C also with a subsequent air-cooling.

Creep tests were performed in two different ways: 1) compression tests at 650 °C with stepwise increasing load (to compare the short-creep behavior of the master alloys with references alloys); and 2) tensional tests with constant load between 100 and 250 MPa (to determine the creep rupture times). The microstructure of the alloys was analyzed in the initial state and after creep conditions with Scanning Electron Microscopy (SEM) and with Transmission Electron Microscopy (TEM). For SEM investigations the samples were electro-polished with A2 electrolyte, at 20 °C and 18 V. TEM samples were prepared by twin-jet-polishing with a solution of ethanol and perchloric acid at -30 °C and 28 V.

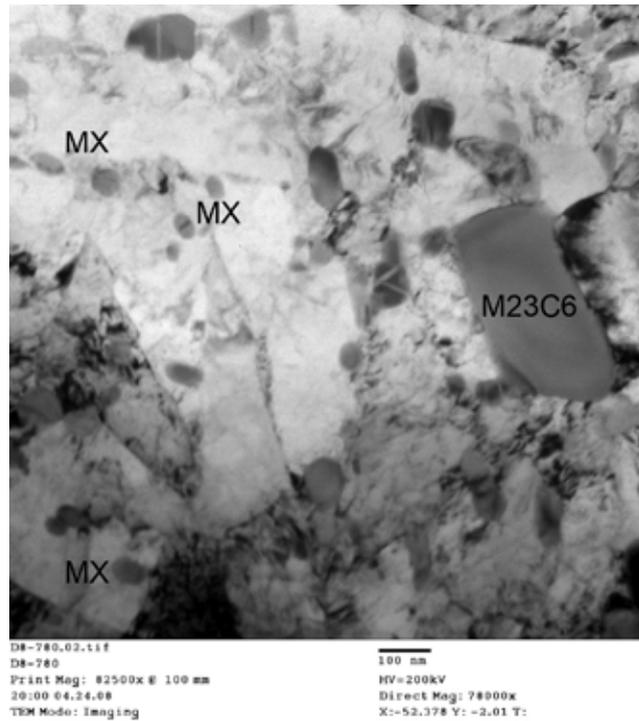
**Microstructure.** Thermo-Calc calculations provide valuable information for adjusting the heat treatment conditions in order to produce stable precipitates and avoid the formation of  $\delta$ -ferrite and undesirable phases. The austenization and the heat treatment temperature after forging was calculated above 1050 °C and below 810 °C respectively for both alloys.

Metallographic investigations of both alloys after heat treatment show a ferritic matrix with the presence of MX carbonitrides and  $M_{23}C_6$  carbides. SEM-EDX qualitative analyses revealed that alloy A1 presents Ta containing MX carbides and  $M_{23}C_6$  carbides of the type (Cr,Fe,W)C. In alloy A1 (due to the high amount of Ta in the starting composition) large TaC formed from the melt (~1  $\mu$ m) are observed. In alloy A2, nanosized MX of the type V(C,N) are present. Due to the relative high amount of B in combination with

relative high amount of W, large W-containing borides (~1-2  $\mu$ m) form from the melt. In alloy A2 the  $M_{23}C_6$  are also of the type (Cr,Fe,W)C, and form principally at the grain boundaries. In Fig. 1 (see next page), a TEM image of alloy A2 after hot-forging and heat treatment showing the presence of nanosized MX carbonitrides (~20 nm) and  $M_{23}C_6$  carbides (~100 nm) is

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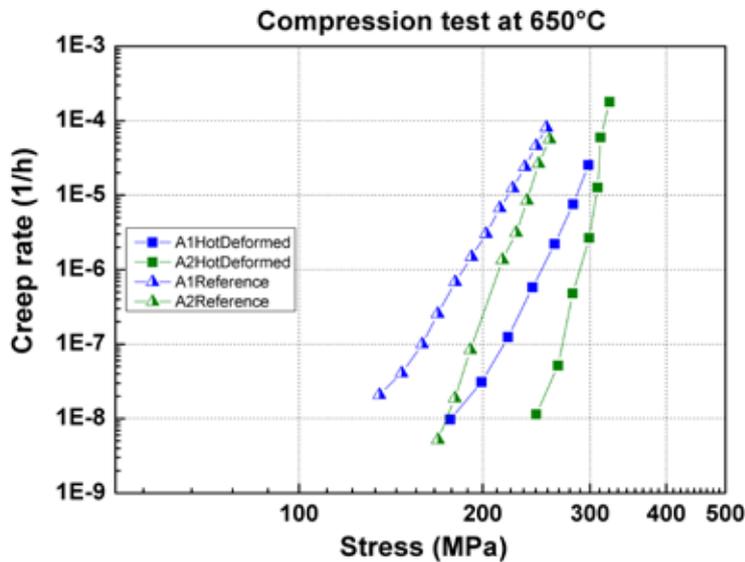
**Fig. 1:** Microstructure of alloy A2 after hot-forging and heat treatment showing nano-sized MX particles and  $M_{23}C_6$  particles.

presented. The phases observed are in accordance with the equilibrium phases calculated with Thermo-Calc (Tab. 2); except for the metastable Laves phase, which precipitates during creep conditions.

**Mechanical properties.** Compression tests were carried out in order to determine the influence of the hot-forging deformation process on the mechanical properties. Compression tests with

	Fe	Cr	W	V	C	N	Co
V(C,N)	0,228	0,345	0,022	78,972	0,036	20,912	-
Laves	29,121	8,294	66,251	-	-	-	0,065
$M_{23}C_6$	14,460	66,497	13,655	0,061	5,068	-	0,107

**Tab. 2:** Composition of precipitates for alloy A2 as calculated with Thermo-Calc.



**Fig. 2:** Results of the compression test at 650 °C for alloys A1 and A2 showing the increment of the creep resistance for the hot-forged alloys.

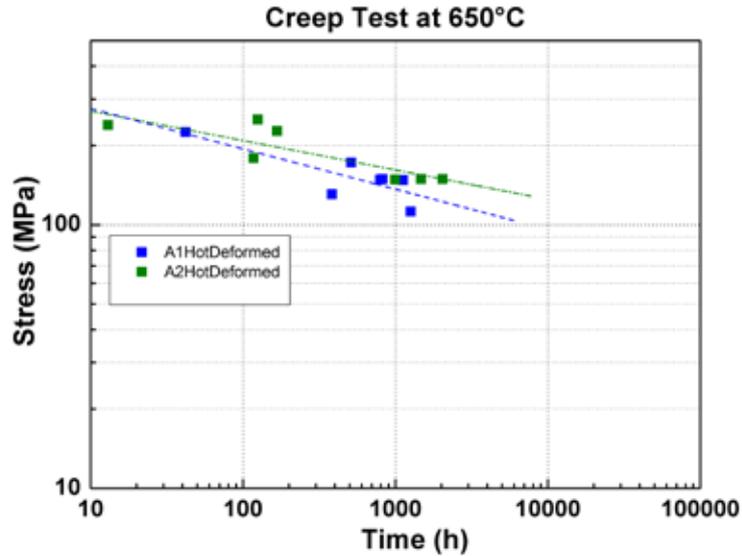


Fig. 3: Creep tests at 650 °C for alloys A1 and A2 showing the increment of the creep resistance in the early stages of creep for the alloy A2.

stepwise increased load at 650°C were used for the determination of the stress dependence of the secondary creep rate. Results of the compression tests (Fig. 2) show an increment of the creep resistance for both alloys A1 and A2 due to the deformation process, i.e. the samples, which were hot-forged, withstand higher loads - between 50 and 70 MPa - for the same creep rate.

Tensile tests at constant loads were carried out for the determination of the creep rupture time. Long term creep tests at 650 °C show the increment of the creep resistance for the alloy A2 compared to alloy A1 (Fig. 3). The better creep resistant of alloy A2 is correlated to the observed and calculated formation of stable nanosized vanadium carbonitrides, which are responsible for the pinning of dislocations during creep.

After creep at 650 °C for 2000 h under a load of 150 MPa, grain growth of  $M_{23}C_6$  carbides is clearly

observed (Fig. 4). On the other hand, scarcely grain growth of the MX carbonitrides is present. The grain growth rate of the  $M_{23}C_6$  is controlled by the Cr-diffusion, which explains the faster growth of this phase compared to the more stable MX carbonitrides.

**Modelling.** Dictra is based on a numerical solution of diffusion equations with local equilibrium at the phase interfaces. The simulation of the coarsening is carried out by considering the growth of the largest particle in a defined calculation cell [3]. The coarsening model considers that the particle size is 1.5 times the average particle size. The local equilibrium at the interface between the particle and the matrix is affected by the interfacial energy. The interfacial energy yields a contribution to the Gibbs energy of the particle as follows:

$$\Delta G_m = \frac{2\sigma V_m}{r}$$

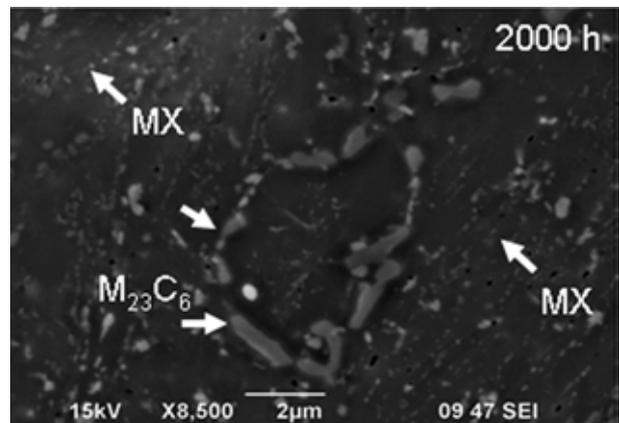
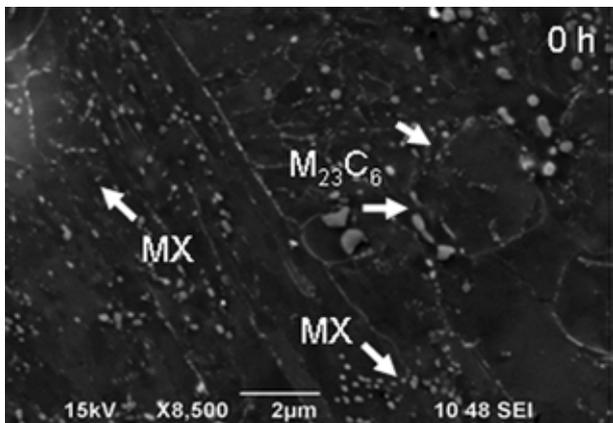
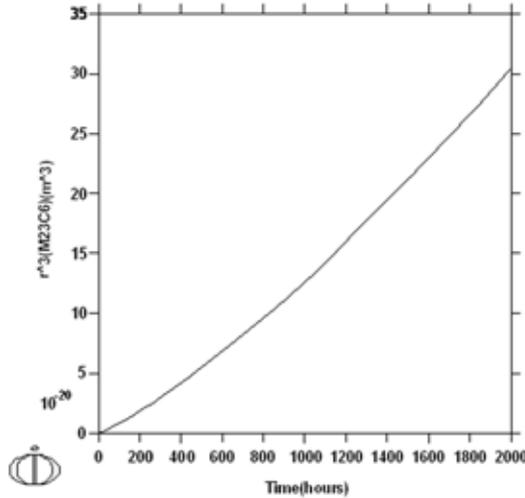
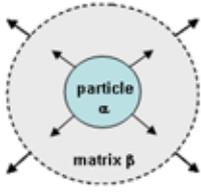


Fig. 4: Microstructure of alloy A2 showing the coarsening of the  $M_{23}C_6$  (at the grain boundaries) and MX particles at the initial stage (left) and after 2000 h creep at 650 °C / 150 MPa (right).



**Fig. 5:** Schematic drawing (left) of the coarsening model employed for the Dictra calculations. The moving phase interface between the carbide particle ( $\alpha$ ) is in local equilibrium with the matrix ( $\beta$ ). At the outer boundary the equilibrium is defined by the average composition in the system. In order to keep the mass balance the cell size is allowed to increase accordingly. Calculated coarsening (right) of  $M_{23}C_6$  for alloy A2 after 2000h creep at 650°C at 150 MPa.

where:  $\sigma$  is the interfacial energy in J/m<sup>2</sup>,  $r$  is the radius in m and  $V_m$  is the molar volume in m<sup>3</sup>/mol substitutional atoms. For the conditions investigated, the model yields the same evolution law as the classical treatment; i.e.:

$$d^3 - d_o^3 = kt$$

where:  $d_o$  is the initial mean particle diameter,  $d$  is the mean particle diameter at the time  $t$  and the parameter  $k$  is the coarsening rate in m<sup>3</sup>/s.

The simulation (database TCFE5 and mobility database MOB2) of the coarsening of  $M_{23}C_6$  was performed with the elements Fe, C, Cr and W, which are the main elements calculated with Thermo-Calc for this phase. It was assumed that the driving force for coarsening stems from the interfacial energy and that the rate of coarsening is controlled by the volume diffusion.

The results of the simulations are shown in Fig. 5 considering a  $\sigma = 0.6$  J/m<sup>2</sup> and  $V_m = 0.6$  m<sup>3</sup>/mol, a temperature of 650 °C and a creep time of 2000 hours. The figure shows the cubic of the average radius of  $M_{23}C_6$  as a function of time. In the simulation the Laves phase is not allowed to form. The results of the simulation are in good agreement with the observed grain sizes determined by quantitative metallography.

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## Combining Experimental and Computational Methods in the Development of Fe<sub>3</sub>Al-based Materials

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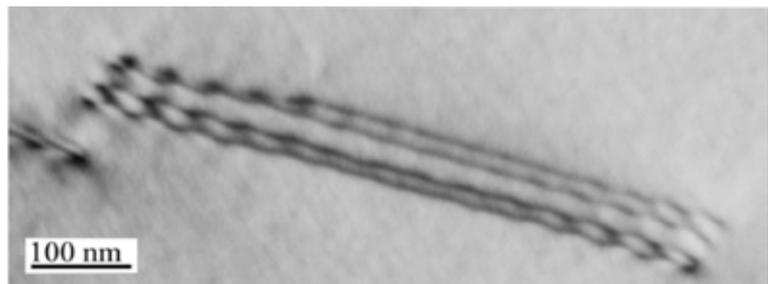
The development of new lightweight materials is crucial for numerous energy-conversion applications in the automotive and aerospace industries. Low-cost and low-density materials operating at higher temperatures ensure a lower fuel consumption and environmentally cleaner and more efficiently produced electricity. Two basic options in materials design and/or functional optimization are the selection of an appropriate chemical composition and the processing of an optimized microstructure. Both characteristics are mutually interlinked and inherently multiscale in nature what make them challenging to study.

We address these fundamental aspects in a promising class of lightweight intermetallics: Fe<sub>3</sub>Al-based alloys exhibiting an excellent high temperature oxidation and sulfidation resistance up to 1000 °C. Therefore, they possess a high potential as a low cost alternative to conventional ferritic, martensitic or austenitic steels [1]. Depending on the Al content Fe-Al alloys exhibit a disordered bcc-, an ordered B2- or a D0<sub>3</sub>-ordered superlattice structure. Most investigations deal with Fe<sub>3</sub>Al-based alloys because of their lower density in comparison to conventional steels, the considerable room temperature ductility (that is quite unusual among intermetallics), as well as the soft magnetic properties.

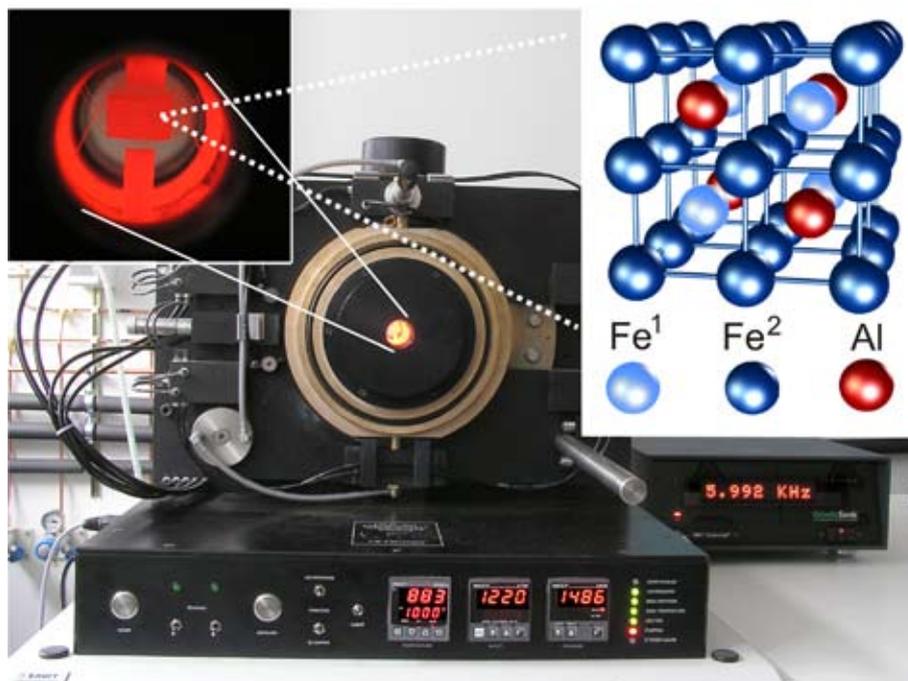
For actual applications as structural materials, however, further improvements of these alloys e.g. via compositional optimization are considered. Specifically, these optimizations should lead to enhanced mechanical properties, such as improved room temperature ductility, or higher strength and creep resistance at temperatures above 600°C. As these alloys have a D0<sub>3</sub>-ordered lattice, the deformation behaviour is rather complex exhibiting single, double, and fourfold dislocations (see an example of the fourfold one in Fig. 1) which are separated by two types of anti-phase boundaries (APB) namely nearest-neighbour- and next-nearest-neighbour APBs. Whether the deformation of Fe<sub>3</sub>Al alloys is dominated by the one or other type of dislocations depends on the temperature, the strain, probably on the APB energy as well as on the degree of order. For the simulation of dislocation motion, or in general the deformation behaviour, the knowledge of the elastic properties is mandatory. Also it is obvious,

that the plastic deformation should be facilitated if the APB energy is low, as less energy is stored in the APBs. The determination of APB energies by the measurement of the distance of separated superpartials in the TEM needs big efforts and may lead to divergent results. With the knowledge of the detailed site preferences and elastic constants APB energies can be calculated and the prediction of alloy compositions with low APB energies should be possible [2,3]. For this, a detailed knowledge of the influence of alloying elements on the physical and mechanical properties is crucial but literature data are hardly available and the experimental determination is very time consuming. The use of new multidisciplinary approaches combining both experimental and theoretical methods is thus very desirable as it promises a remarkable reduction of the experimental effort and corresponding costs.

Theoretical *ab initio* calculations based on fundamental quantum-mechanical laws are nowadays increasingly used [4,5] to accurately predict material properties without any empirical input, solely from the knowledge of the atomic composition and/or structure. Basic mechanical and physical properties, like crystal structure and single crystal elastic constants, can be reliably calculated at zero temperature using density functional theory (DFT) [6,7]. The calculated materials characteristics gathered at the atomistic level and our deeper understanding of the underlying processes can be effectively combined with advanced experimental techniques in order to design new materials with desired properties. Regarding specifically the iron-aluminides, (i) the site preference of the alloying elements and (ii) changes in the Young's modulus have been determined in this study. In order to analyse compositional trends of both thermodynamic and elastic properties of Fe<sub>3</sub>Al-based



**Fig. 1** TEM micrograph of a fourfold dislocation in D0<sub>3</sub>-ordered as-heated Fe-26Al after 0.6 % tensile deformation at room temperature.



**Fig. 2** The Young's moduli have been determined by the impulse excitation technique. For this samples with dimensions of  $70 \times 7 \times 4$  mm have been cut by spark erosion from vacuum induction melted ingots of several  $\text{Fe}_3\text{Al-X}$  alloys. The temperature dependent values have been obtained by a fully automated Young's modulus measurement device (Grindosonic) at a heating rate of 100 K per hour at every K in argon atmosphere. The right-hand upper inset shows the  $\text{DO}_3$ -based supercell with 8  $\text{Fe}^2$  and 4  $\text{Fe}^1$  positions (dark and light blue) and four Al sites (red).

single-crystalline compounds, the supercell approach with a 16-atomic supercell derived from the  $\text{DO}_3$  unit cell of  $\text{Fe}_3\text{Al}$  was employed (see the inset in Fig. 2).

The  $\text{DO}_3$  unit cell consists of two iron sublattices,  $\text{Fe}^1$  and  $\text{Fe}^2$ , the latter one having a twice higher number of Fe atoms than the former one, and one Al sublattice. As the total number of atoms in the computational supercell is 16, the minimum concentration of the substituents when replacing a single Fe or Al atom is 1/16 or 6.25 at. %.

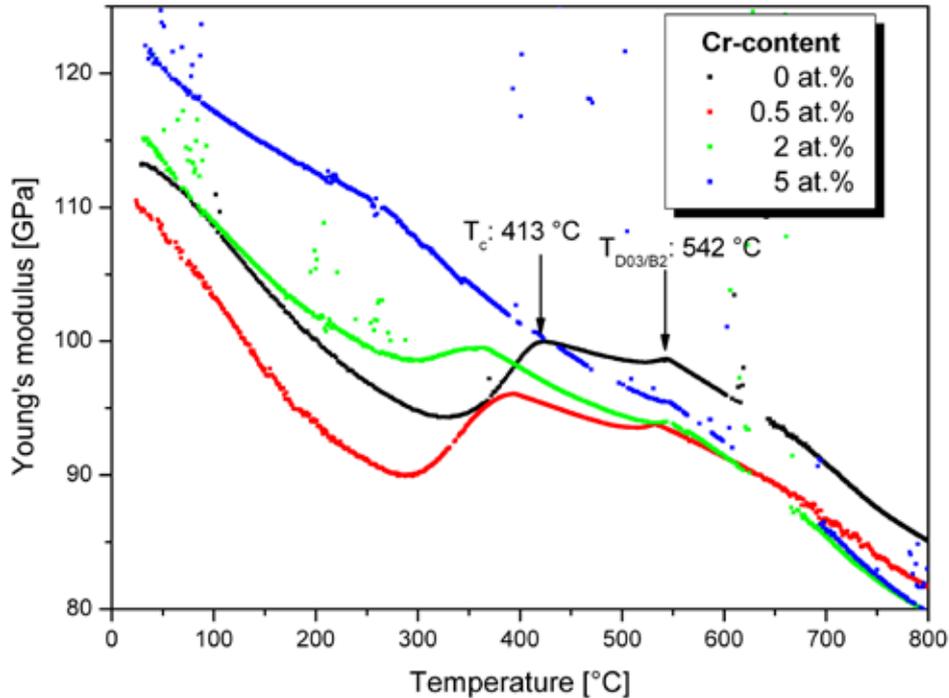
The integral polycrystalline Young's modulus can also be determined theoretically from a multi-physics combination of (i) the *ab initio* single-crystal elastic constants [8] and (ii) continuum linear-elasticity homogenization (Hershey's homogenization technique [9]). The *ab initio* calculations have been performed for  $T=0\text{K}$  conditions but the qualitative validity of the predicted metallurgical trends can be extended also to finite temperatures.

The theoretical research has been complemented with measurements of the Young's modulus. By means of the impulse excitation method, experiments have been performed at 77 K and continuously from 293 K to 1273 K, i.e., in the relevant temperature range of possible applications of  $\text{Fe}_3\text{Al}$ -based alloys (see Fig. 2). Both the calculations and measurements have been performed for a selected group of substituents (Ti, W, V, Cr, Si) exhibiting a solubility in  $\text{Fe}_3\text{Al}$  at least as high as 2 at. %.

**Results.** The site preference of the substituents has been studied by calculating the defect formation energy of ordered  $\text{Fe}_3\text{Al}$ -based compounds with a substituent X located on either one of the two iron sublattices,  $\text{Fe}^1$  or  $\text{Fe}^2$ , or replacing one Al atom (see the right inset in Fig. 2). Both the internal and external degrees of freedom, including different magnetic states, were optimized with respect to the total energy.

In agreement with previously published experimental data (see e.g. [10]) the obtained results clearly show a strong preference of Ti, W, and V for the  $\text{Fe}^1$  sublattice. The preference has been found weaker in case of Cr, making the  $\text{Fe}^2$  substitution also possible especially at elevated temperatures, whereas Si atoms show a strong site preference for the Al sublattice.

Motivated by the use of  $\text{Fe}_3\text{Al}$ -based alloys as structural materials, the lattice constant change upon the substitution has been also theoretically determined. As the experimental measurements clearly show linear trends in the lattice parameter dependence as a function of the solute content in the low concentration region, the values theoretically predicted for 6.25 at. % can be directly compared to the measurements for other concentrations. In agreement with the experimental data [11,12], the *ab initio* calculations predict an expansion of the lattice for Ti and W but a contraction in case of V, Cr, and Si.



**Fig. 3** Temperature dependence of the Young's modulus for ternary  $Fe_3Al$ -Cr alloys. For the binary alloy the Curie temperature  $T_c$  and the  $D0_3/B2$  transition temperature  $T_{D03/B2}$  are marked by arrows. Between 600 and 700 °C the damping of phonons in the lattice is often so high that the resonance frequency can not be detected.

In order to identify elements that may possibly increase the strength of the  $Fe_3Al$ -based materials, the change in the homogenized polycrystalline Young's modulus  $E$  due to solutes has been determined. The calculations predict an increase in the moduli in case of all studied alloying elements, i.e. Ti, V, W, Cr, and Si. Assuming a linear increase in  $E$  with solute atom concentration, the actual changes  $\Delta E^{theo}$  obtained due to 6.25 at.% of the alloying element with respect to pure  $Fe_3Al$  are summarized in Tab. 1 together with corresponding experimental data.

To cross-check the validity of the *ab initio* prediction, the influence of the same five ternary elements on the Young's modulus has been investigated also experimentally. To perform measurements under conditions close to those of the *ab initio* calculations, the Young's moduli have been at first determined at 77 K. In agreement with the theoretical predictions, all the studied alloying elements increase the Young's modulus with respect to that of pure  $Fe_3Al$  in the

(GPa/at.%)	V	Cr	W	Si	Ti
$\Delta E^{theo}$	6.8	3.1	9.3	5.5	3.9
$\Delta E^{exp}$	7.5	1.2	13.2	9.0	26.6

**Tab. 1** The increase in the homogenized polycrystalline Young's modulus  $E$  (in GPa per at.% with respect to  $Fe_3Al$ ) in  $Fe_3Al$ - $X$  ( $X=V, Cr, W, Si, Ti$ ) as theoretically predicted ( $\Delta E^{theo}$ ) for 6.25 at.% of solutes and from a linear fit to the experimental data obtained for different solute contents ( $\Delta E^{exp}$ ).

concentration range above 1-2 at.% (see  $\Delta E^{exp}$  in Tab. 1). A similar increase has been detected also at room temperature.

A highly unexpected elastic effect in the Young's modulus compositional trend was observed in some  $Fe_3Al$ -Ti and  $Fe_3Al$ -W alloys for small additions of the solute atoms: The expected linear trends (e.g. in iron [13]) have been not observed there. The Young's modulus has been found even *decreasing* in some cases in the concentration range below 1 at.% due to these solute atoms. As almost no literature data are available on the concentration dependence of the elastic properties for low solute concentrations in case of binary systems and even less for ternary systems, this interesting effect remains unexplained and will be a part of future investigations.

The temperature dependence of the Young's modulus is discussed exemplarily for the  $Fe_3Al$ -Cr alloys (see Fig. 3). The slopes of the curves are characterized first by a decrease to a local minimum and then an increase up to the Curie temperature,  $T_c$ . Between  $T_c$  and the  $D0_3/B2$  transition temperature ( $T_{D03/B2}$ ) the curves decline rather slow or even incline again in the vicinity of the order transition temperature (see Fig. 3). Above  $T_{D03/B2}$  the Young's modulus drops almost linearly with increasing temperature as it is the case for most single phase alloys. The local minimum of the Young's modulus below  $T_c$  can be explained by magnetostrictive effects and is well documented for other ferromagnetic materials e.g. Ni-based alloys exhibiting an Invar-like behaviour [14].

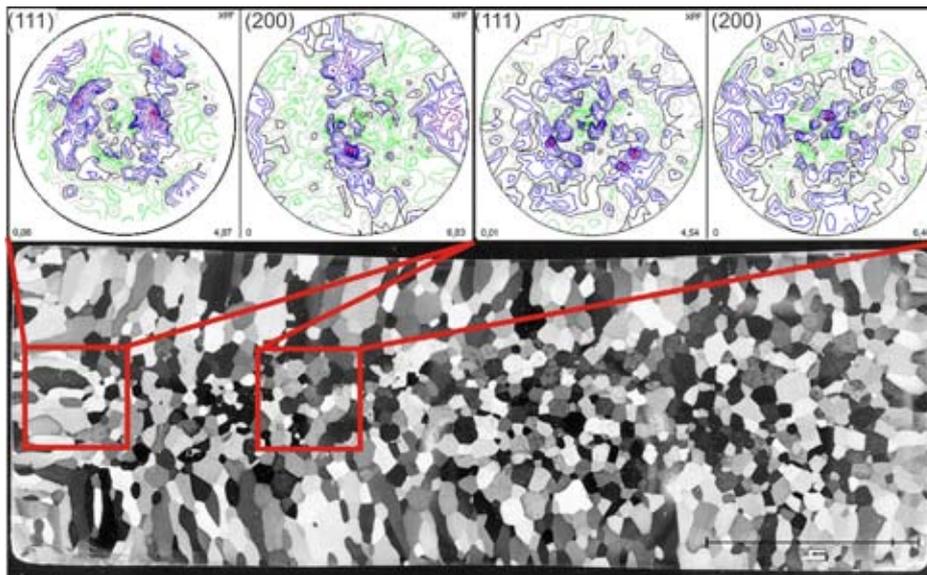


Fig. 4 Pole figures and corresponding micrographs of the 1 cm<sup>3</sup> Fe<sub>3</sub>Al sample (courtesy of Prof. Raabe and Dr. Zaeferrer, MPIE).

At room temperature, the measurements were performed on different sample directions and exhibit a considerable anisotropy. This may result from crystallographic textures of the as cast microstructure which can be rather strong for some Fe-Al based alloys (see Fig. 4). As the deviations from an idealized microstructure (expected by Hershey's homogenization) have not been incorporated in the theoretical modelling, the texture variations can explain differences between the predicted and measured data.

The presented results show that very promising multi-disciplinary research combining both theoretical *ab initio* and experimental techniques has been performed and exemplified for the development of improved Fe<sub>3</sub>Al-based materials. The Young's moduli experimentally determined at elevated as well as liquid-nitrogen temperatures were complemented by T=0K *ab initio* calculations of thermodynamic, structural, and elastic properties. Excellent qualitative agreement between the predicted and measured data has been found. Advanced methodological development is planned in order to (i) take strong textures of the Fe<sub>3</sub>Al alloys into account and (ii) to explain the unexpected trends at low-concentrations for W and Ti.

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## Processing and Mechanical Properties of Ultrafine Grained Dual-Phase Steels

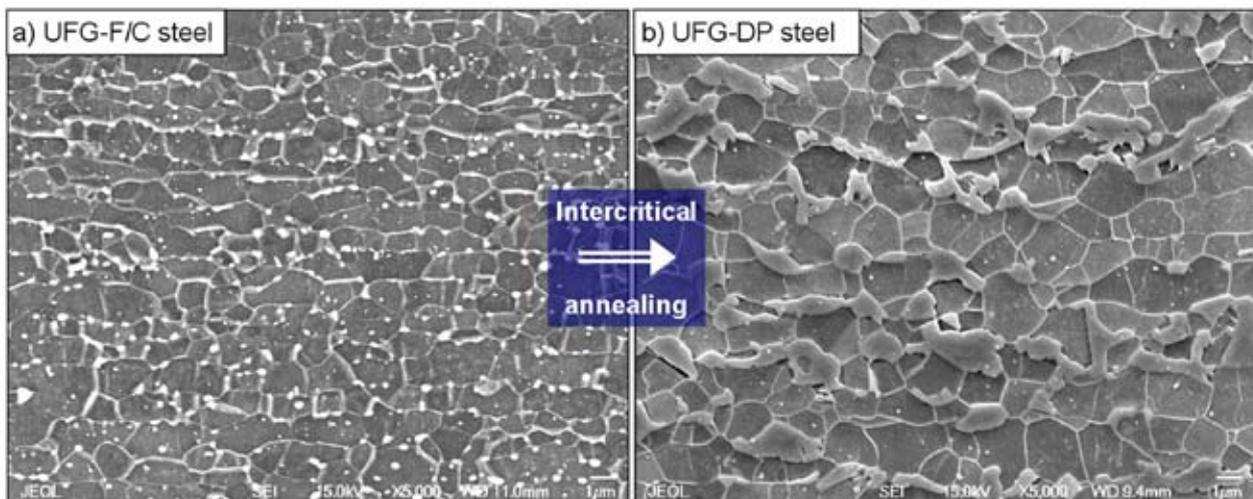
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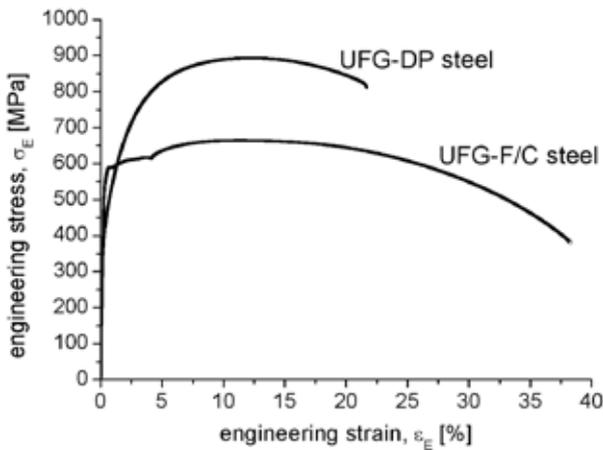
Grain refinement is a well-studied method for property optimization in steels as it is the only strengthening mechanism that enhances strength and toughness simultaneously. In recent years, grain refinement down to micron scale has attracted the interest of scientists and engineers due to the development of new grain refinement techniques [1]. Yet, the main drawback of ultrafine grained (UFG) steels is their restricted tensile ductility due to a very low strain hardening rate. One attempt to improve the strain hardening rate is the introduction of martensite as a hard second phase into the soft ferrite matrix [2,3]. Such dual phase (DP) steels are known to exhibit a high initial strain hardening rate as a result of the large amount of mobile dislocations and to internal stresses introduced during the martensitic phase transformation [4]. DP steels are widely used for automotive applications as they combine high strength with good ductility. Regarding the increasing demand for fuel economy and occupant safety, it is of particular importance to further improve the mechanical properties without raising alloying costs or allowing a decrease in ductility. Grain refinement of DP steels was shown to increase both yield strength and tensile strength, whereas strain hardening rate, uniform and total elongation are hardly affected [5,6]. For these reasons, the motivation of this project is two-fold. One is the improvement of the ductility of ultrafine grained steels, the other is the increase in strength of dual-phase steels.

**Methods.** The investigated material is a plain 0.15%C-1.5%Mn-0.3%Si-0.03%Al steel. In a recently published paper [7], it was demonstrated that a certain Mn content is necessary for the formation of martensite during quenching. For materials processing, a large scale 2.5 MN hot press located at the Max-Planck-Institut für Eisenforschung was used [8]. It was shown in previous projects [9,10] that grain refinement to  $\mu\text{m}$  scale can be achieved by large strain warm deformation. By exerting a four-step flat compression series at 550°C with a total strain of 1.6, an ultrafine grained ferrite matrix with homogeneously distributed spheroidized cementite particles (UFG-F/C steel) was obtained. The final ferrite/martensite dual phase structure was produced by an intercritical annealing treatment in the ferrite/austenite two phase region followed by quenching. For microstructure and texture investigations, a high resolution secondary electron microscope (SEM) equipped with field emission gun (FEG) and energy-dispersive X-ray spectrometer (EDS) was used.

**Microstructure and mechanical properties.** The UFG-F/C steel consists of a ferrite matrix with an average grain size of 0.9  $\mu\text{m}$  (Fig. 1a). Fine cementite particles (2-3 vol.%) are distributed homogeneously mostly along ferrite grain boundaries and to a less extent inside ferrite grains. After intercritical annealing at 730°C for 1 min, the average ferrite grain size increases slightly to 1.2  $\mu\text{m}$  and as can be seen from Fig. 1b, cementite is replaced completely by



**Fig. 1:** Microstructure of the ultrafine grained (UFG) ferrite/cementite steel obtained after large strain warm deformation (a) and of the ultrafine grained ferrite/martensite dual phase steel obtained after large strain warm deformation and subsequent intercritical annealing (b).



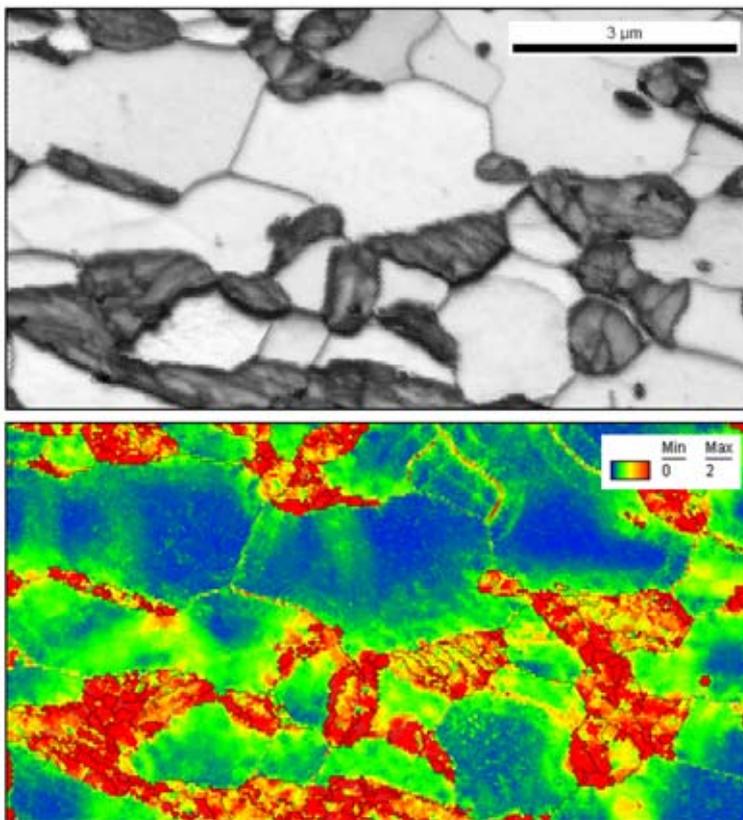
**Fig. 2:** Mechanical properties of the ultrafine grained (UFG) ferrite/cementite steel and the ultrafine grained ferrite/martensite dual phase steel.

martensite which makes up a fraction of 27 vol.%. By EBSD investigations, a retained austenite volume fraction of around 3 vol.% was determined.

The stress-strain curves corresponding to the microstructures in Fig. 1 are shown in Fig. 2. The UFG-F/C steel exhibits a relatively high yield strength, a large Lüders strain and a very low strain hardening rate. In contrast, the UFG-DP steel exhibits a very high initial strain hardening rate and continuous

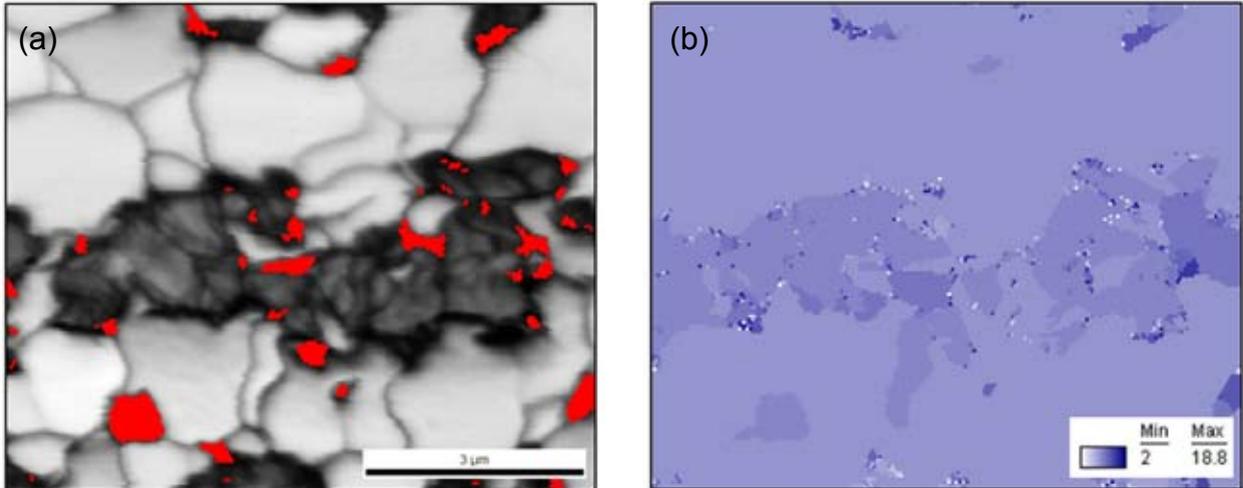
yielding. Due to the high strain hardening rate, tensile strength is increased to 893 MPa, compared to 665 MPa for the UFG-F/C steel. On the other hand, uniform elongation is not significantly improved (11.3% for UFG-DP compared to 10.6% for UFG-F/C) and total elongation to fracture is reduced.

There is broad consensus that the continuous yielding and the high initial strain hardening rate are a consequence of the austenite to martensite transformation which involves a volume expansion. The strains produced by the transformation result in internal stresses in the surrounding ferrite [11]. These internal stresses are assumed to facilitate plastic flow and hence, lower the elastic limit. Furthermore, the volume change induces plastic deformation of adjacent ferrite grains and, therefore, is believed to create a high density of unpinned dislocations in the vicinity of martensite [12,13] as was studied by transmission electron microscopy (TEM) [14,15]. The enhanced dislocation densities surrounding martensite were often documented for coarse grained DP steels using TEM. However, TEM investigations have the shortcoming that only a small area can be observed and that sample preparation can create defects or recovery of the microstructure. By using high-resolution EBSD, crystallographic data of a larger area can be investigated. Changes in lattice orientation yield information about local geometrically necessary dislocation accumulations. Fig. 3a shows an Image Quality map of the UFG-DP steel, where martensite appears black due to the high degree of lattice imperfection. In Fig. 3b, the corresponding Kernel average misorientation map is depicted. This misorientation measure quantifies the average misorientation around a measurement point with respect to all its neighbouring points at a defined radius (here 6 times the step size of 50 nm) and within a certain threshold (here 2°). This study clearly reveals distinct orientation gradients around martensite particles, which reflect high dislocation densities at the ferrite/martensite interface. Orientation gradients are even visible in very small grains <1 μm. On the other hand, ferrite/ferrite grain boundaries do not show enhanced dislocation accumulation. The inhomogeneous distribution of additional dislocations introduced by the martensitic phase transformation is also visualized in this map. Hence, the earlier TEM investigations are verified by the EBSD study as valid also for ultrafine ferrite grains.



**Fig. 3:** High-resolution EBSD study revealing high local average misorientations around the martensite particles. The local average misorientations as expressed by the kernel average misorientation measure (KAM) can be translated into high local densities of geometrically necessary dislocations (top: image quality map, bottom: Kernel average misorientation map).

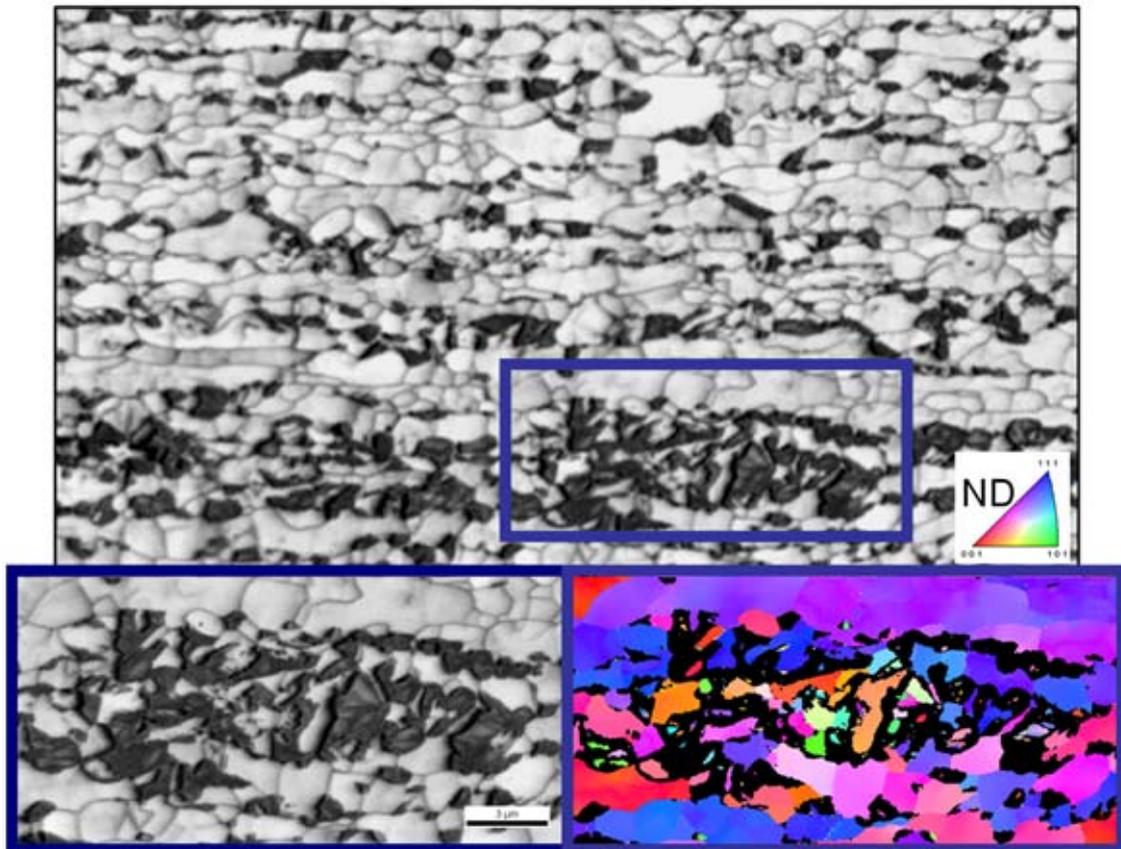
**Hardenability.** Primarily, hardenability is a function of carbon content in austenite.



**Fig. 4:** Retained austenite marked in red in ultrafine grained dual phase steel (UFG-DP) (a) and Mn enrichment in martensite (b).

However, Mn also plays an important role in controlling hardenability. Mn partitions preferentially to austenite and increases its hardenability, i.e. it lowers the critical cooling rate for martensite formation. The Mn enrichment in martensite can be visualized by combined EBSD and energy-dispersive spectrometry (EDS). Fig. 4a shows the Image Quality map of the UFG-DP steel and the position of retained austenite (marked in red). In Fig. 4b the corresponding distribution of Mn is presented in

a semi-quantitative way. The Mn concentration is normalized for each grain. One can clearly correlate higher Mn concentrations with martensite and retained austenite. It was shown in a previous study [16] that large strain warm deformation leads to Mn enrichment in cementite. The present observations imply that the high Mn concentration is passed on to austenite and is later inherited by martensite. It can be assumed that through the additional Mn enrichment in cementite during large strain warm



**Fig. 5:** Because of the high hardenability, the cooling rate can be decreased in order to achieve a partial break-up of martensite bands. The epitaxial ferrite can be identified from the inverse pole figure maps due to its random lattice orientation.

deformation the austenite has a higher Mn content than it would have if it was formed from a conventional ferrite-pearlite structure. Therefore, an enhanced hardenability can be anticipated. Fig. 5 shows an EBSD scan of a sample cooled at 20 K/s from 730°C. Even at that relatively low cooling rate, the austenite-to-martensite transformation is complete. It is important to note that this low cooling rate can be applied even without the addition of hardenability enhancing alloying elements. Slower cooling rates are generally preferred in industrial applications as they yield better strength-ductility combinations. Another important aspect is that due to a slow cooling rate, epitaxial ferrite grains grow inside of larger martensite particles (Fig. 5). Here, the newly grown ferrite grains are easily identified by their random lattice orientation. It is seen that the formation of epitaxial ferrite grains leads to a partial break-up of martensite banded structures and large martensite particles. Martensite bands are generally undesired due to a low crack propagation resistance. Large martensite particles generally deteriorate formability. Hence, adjusting the cooling rate is particularly important for property optimization of the investigated UFG-DP steel.

It was shown that ultrafine grained dual phase steels can be produced efficiently by use of large strain warm deformation and subsequent intercritical annealing. The characteristic low strain hardening rate of ultrafine grained steels is overcome by the introduction of martensite as a hard second phase. The hardenability of the ultrafine grained dual phase steel is enhanced due to the Mn enrichment in cementite during large strain warm deformation which is inherited by austenite.

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## Using *Ab Initio* Calculations in Designing bcc Mg-Li Alloys for Ultra Light-Weight Applications

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*Ab initio* calculations are becoming increasingly useful to engineers interested in designing new alloys because these calculations are able to accurately predict basic material properties [1-4] only knowing the atomic composition of the material. Based on theoretically determined properties, engineering parameters like the ratio of bulk modulus over shear modulus ( $B/G$ ) and the ratio of Young's modulus over mass density ( $Y/\rho$ ) are calculated. The former ratio can indicate either brittle or ductile behavior of studied materials. Specifically the bulk modulus  $B$  is difficult and expensive to measure but can be quite easily calculated. Hence, a multi-disciplinary combination of both experimental and theoretical approaches is the optimum research strategy and we have applied it when designing magnesium-lithium alloys for ultra light-weight applications.

Magnesium is a common, lightweight metal that could potentially be more widely used as a structural material in applications where weight savings is crucial. The use of wrought Mg and Mg-alloys is limited in large scale manufacturing operations for two reasons (i) a lack of room temperature ductility, and (ii) the formation of sharp basal or near basal deformation textures (i.e. the tendency of grains to rotate to preferred orientations during deformation) that lead to strong anisotropy in the material.

In short, Mg and Mg-alloys are difficult to deform at room temperature, and once formed they have undesirable mechanical properties.

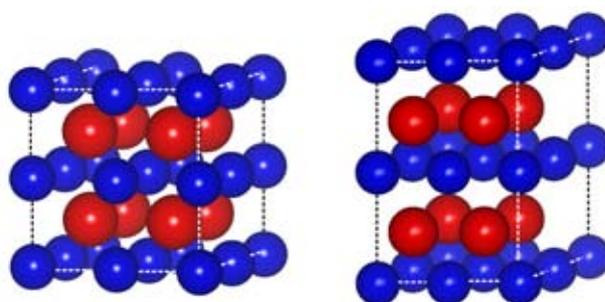
The absence of room temperature ductility and the evolution of strong deformation textures are inherent limitations of the hcp crystal structure that to date have not been completely overcome. Changing the crystal structure from hcp to either face centered cubic (fcc) or body centered cubic (bcc) would make Mg more attractive from a manufacturing point of view. Alloying Mg with as little as 30 at.% Li results in a stable bcc Mg-Li alloy at room temperature. Not only does Li stabilize the bcc crystal structure; it also has the advantage of further decreasing the density of the Mg alloy since lithium is the lightest known metal ( $\rho^{\text{Li}} = 0.58 \text{ g/cm}^3$ ,  $\rho^{\text{Mg}} = 1.74 \text{ g/cm}^3$ ). The resulting Mg-Li alloys are therefore among the lightest possible metallic alloys, some are even less dense than water.

In order to experimentally investigate various structural phases of Mg-Li alloys, it is necessary to first manufacture the alloys and then determine the relevant mechanical and physical properties. Undertaking such a task is impossible if the necessary infrastructure is not available and even with the required set-up, casting and then testing Mg-Li alloys will be both time consuming and expensive.

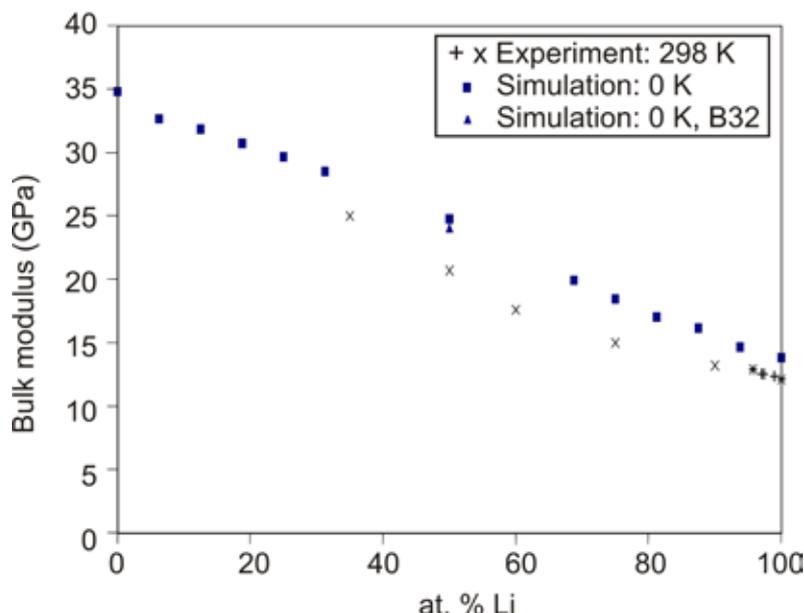
A great advantage offered by computational material science tools like first-principle calculations is the ability to reasonably estimate certain key mechanical and physical properties prior to any experimental work. Another advantage offered by quantum-mechanical calculations is the ability, with relative ease, to access properties that can be experimentally difficult to determine.

Here we report on first-principles calculations of single crystal elastic constants of bcc Mg-Li alloys [3,4] and compare the results with available experimental data. The single crystal elastic constants for bcc-Mg, bcc-Li, and 15 different substitutional alloys were calculated using density-functional theory [5,6] (DFT). The binary alloys were modeled as  $2 \times 2 \times 2$  elementary cubic cells that contained 16 atoms. Alloy compositions were varied by systematically replacing Mg and Li atoms and the resulting compositions ranged from 6.25 at.% Li to 93.75 at.% Li (see Fig. 1).

The calculated metallurgical trends in the single-crystalline bulk modulus of bcc Mg-Li alloys are



**Fig. 1** The  $2 \times 2 \times 2$  16-atomic supercells used in the calculations, (i) for the cubic phase on the left side, (ii) and the hexagonal close-packed one on the right. Odd and even atomic layers are distinguished by different colors for sake of clarity.



**Fig. 2** Simulated and experimental hydrostatic bulk modulus  $B$  for bcc Mg-Li alloys. (Experimental data: + Trivisonno and Smith [7]; x Lynch and Edwards [8].)

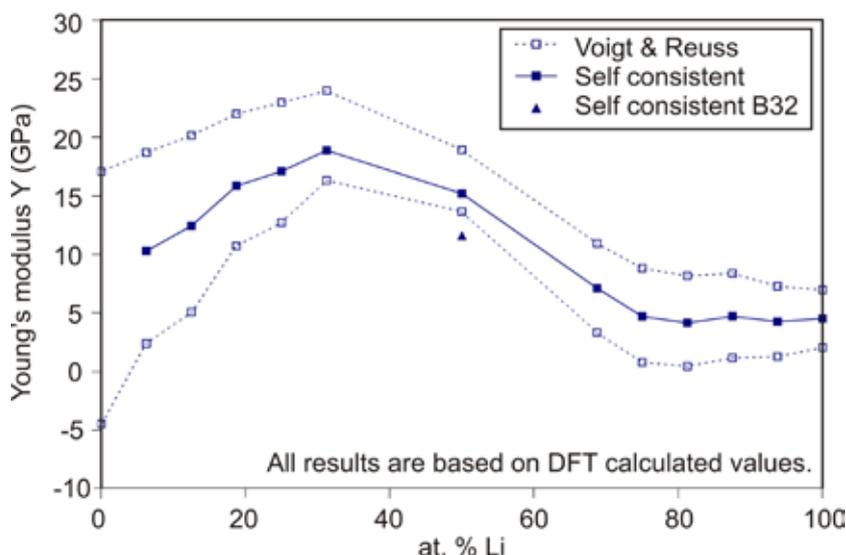
shown in Fig. 2. A good agreement between the theoretical prediction and available experimental data can be seen.

**Polycrystalline elastic moduli.** The DFT based approach can directly calculate single crystal elastic constants, but most engineering materials are polycrystalline in nature. The polycrystalline elastic constants must then be estimated from single crystal elastic constants using various homogenization schemes.

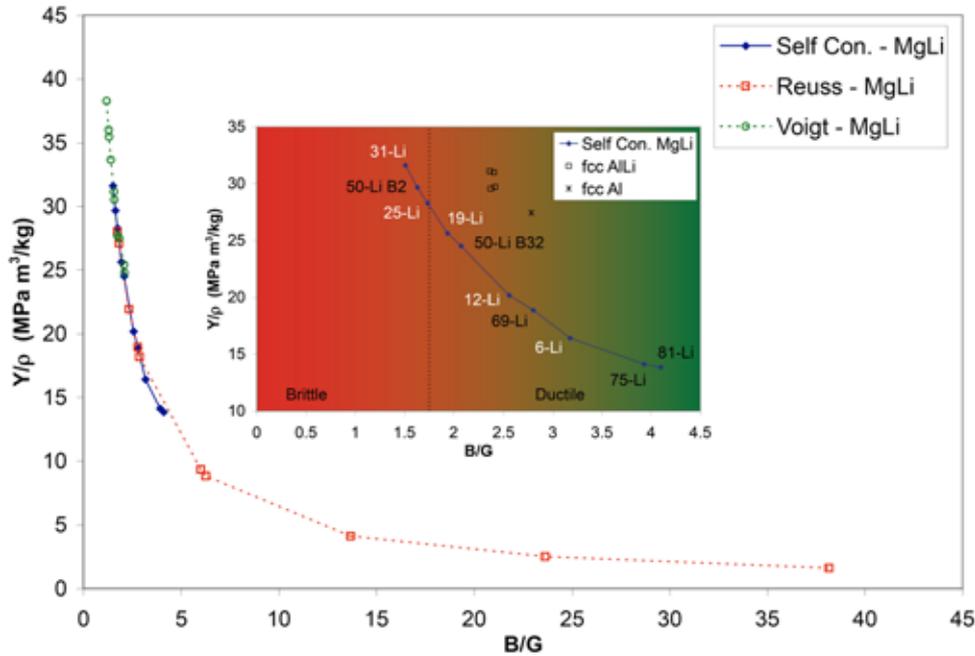
An upper (or Voigt [9]) and lower (or Reuss [10]) bound on the polycrystalline shear and bulk modulus as well as a self-consistent estimate (Hershey [11,12])

can be calculated from the single crystal elastic constants. Once the homogenized values of the shear modulus  $G$  and bulk modulus  $B$  are known, the Young's modulus ( $Y$ ) of a polycrystalline alloy that is elastically isotropic can be calculated using standard elasticity relationships. The calculated compositional trend in the Young's modulus  $Y$  for bcc Mg-Li alloys are shown in Fig. 3. A similar trend was obtained also for the polycrystalline shear modulus  $G$ .

The results in Fig. 2 show that the bulk modulus increases almost 150% in a linear fashion from a minimum at bcc Li to a maximum at bcc Mg. The compositional dependence of both  $Y$  and  $G$ , on the other hand, is highly non-linear.



**Fig. 3** *Ab initio* predicted polycrystalline Young's modulus based on calculations of 15 different ordered Mg-Li compounds [3,4]. Specifically for the 50 at% Li, two different compounds were studied with B2 and B32 structures in order to estimate an error bar of the theoretically predicted values.



**Fig. 4** Ashby map of  $Y/\rho$  vs.  $B/G$  for bcc Mg-Li alloys and fcc Al-Li alloys. ( $Y$  = Young's modulus,  $B$  = hydrostatic bulk modulus,  $G$  = shear modulus.) All modulus values are representative of polycrystalline materials with a random texture. The range of values corresponding to the ductile behaviour is marked by the greenish background and the brittle one by the reddish one in the inset. Systems with different chemical compositions are identified by their Li content in the inset, i.e. 12-Li means 12 at.% Li, and specifically in case of 50 at.% Li compounds, also the structure type is listed, i.e. B2 or B32.

The softest Mg-Li alloys contain large amounts of Li, while the stiffest alloys contain around 30 at.% Li. In addition, the self-consistent homogenization approach predicts that pure bcc-Mg will be mechanically unstable (i.e. bcc-Mg will undergo a deformation induced phase transformation).

**Engineering parameters.** To date, there is no known approach by which all the properties of an alloy can be optimized. Instead, improvement in one area (for example stiffness) often comes at the price of a reduced property in another area (like ductility). Therefore, alloys are designed to fulfill a certain set of prescribed criteria. In order to evaluate which alloys will best fulfill the necessary criteria, two separate engineering ratios, which only use DFT calculated quantities, are employed. The first is the ratio between bulk modulus and shear modulus ( $B/G$ ). This ratio has been correlated to the brittle and ductile behaviour [13].

The second is the ratio of Young's modulus to density ( $Y/\rho$ ) or specific modulus. This ratio quantifies the stiffness of a material per unit weight, an important engineering quantity for light-weight materials.

To provide perspective, the DFT predicted properties of bcc Mg-Li alloys have been plotted with DFT predicted properties of fcc Al-Li [14,15], one of the best commercially available light-weight structural alloys. A direct comparison between bcc Mg-Li alloys and fcc Al-Li alloys can best be made by plotting an

Ashby map, i.e.,  $B/G$  vs. specific modulus. The Voigt, Reuss, and self-consistent values for Mg-Li alloys with 6-80 at.% Li have been plotted in Fig. 4.

An interesting and unexpected result of this plot is that the Reuss, Voigt, and self-consistent values no longer form a lower bound, an upper bound, and an approximate average, but virtually coincide on a single (and for this alloy system) universal *master curve*. Also, the two local structures considered at 50 at.% Li lie on the same line (see inset of Fig. 4). Specifically, the Voigt values define the upper bound on  $Y/\rho$ , the Reuss values define the upper bound on  $B/G$ , and the self-consistent values fall in the region where the Voigt and Reuss values overlap.

The inset graph in Fig. 4 focuses on the self-consistent homogenization part of the master curve. The composition dependence of the self-consistent curve can be explained as follows. The most ductile alloy and least stiff alloy has 81 at.% Li. Decreasing the Li content (75, 69, 50-B32, and 50-B2 at.% Li) results in stiffer, less ductile alloys. The stiffest, least ductile alloy has 31 at.% Li. Further decreasing the Li content (25, 19, 12, 6 at.% Li) results in alloys with more ductility and less stiffness.

The discovery of the universal character of the master curve is consistent with an intrinsic constraint in alloy design concept: optimization of one property often comes at the expense of another property. For the bcc Mg-Li alloys, it can be seen that an increase in



specific modulus is accompanied by a corresponding tendency towards more brittle behaviour (i.e. a decrease in the B/G ratio). The same trend can also be seen in the Al-Li alloys when they are compared with pure fcc Al. An interesting consequence of this analysis is that it does not appear to be possible to increase both  $Y/\rho$  and B/G by changing only the composition or local order of this binary alloy. Therefore, we plan to extend this study to ternary (quaternary) alloys.

The B/G vs.  $Y/\rho$  plot can be used to identify alloys with a combination of properties that are needed in a light-weight structural alloy. Bcc Mg-Li alloys with 30-50 at.% Li have properties that could potentially rival or better that of Al-Li alloys. However, the predicted B/G ratio for these Mg-Li alloys is lower than that predicted for Al-Li alloys. Despite the lower B/G ratio, the increased specific modulus of certain bcc Mg-Li alloys makes this an interesting material system that could offer potential weight savings in the future.

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# High Temperature B2-Ordered NiAl-Based Alloys Reinforced by Refractory Metals for Advanced Energy Conversion Technologies

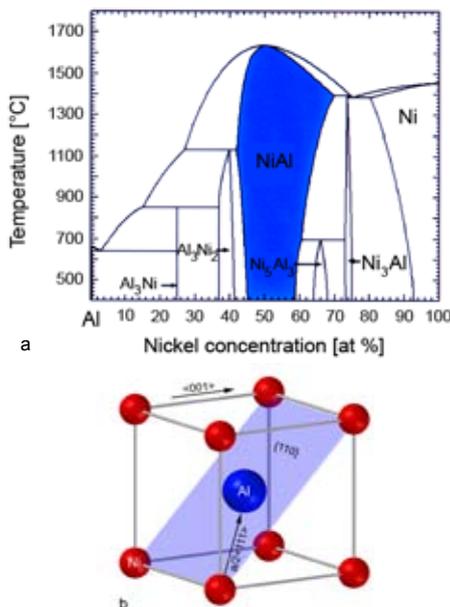
G. Frommeyer, R. Rablbauer\*

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The high melting point aluminides of the iron group metals, such as NiAl, CoAl, or FeAl exhibit potential capacity for high temperature applications. These materials are predestined for components in energy conversion systems such as stationary gas turbines of power plants, internal combustion engines, heat exchangers and jet engines operating at high temperatures and in severe corrosive environments. Higher efficiencies will lead to a significant reduction in fuel consumption and a considerable decrease in exhaust gas emissions. High temperature resistant alloys based on the intermetallic compound NiAl alloyed with the refractory metals chromium, molybdenum or rhenium may fulfill the technological requirements as materials for prospective energy conversion technology operating up to 1300 °C [1-3].

**Structure and Properties of Single Phase NiAl.** The intermetallic compound NiAl with B2 superlattice structure exhibits superior properties, such as high elastic stiffness, Young modulus of about  $E = 180 \text{ GPa}$  [4], good thermal conductivity of  $\lambda_{\text{ther}} = 92 \text{ W m}^{-1} \text{ K}^{-1}$  [5], low density of  $\rho = 5.9 \text{ g cm}^{-3}$ , and excellent oxidation resistance up to 1300 °C [6,7]. The high melting point of  $T_{\text{sm}} = 1674 \text{ °C}$  [8] increases the effective operating temperatures of engine components in comparison with conventional nickel- or cobalt based superalloys.

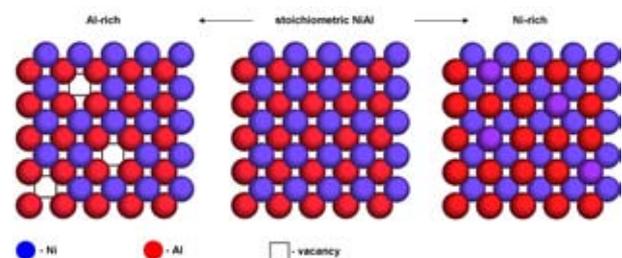
The binary phase diagram of Ni-Al is presented in Fig. 1a [9]. The striking feature is the extended



**Fig. 1:** Binary Al-Ni phase diagram (a) and B2-ordered b.c.c. unit cell of NiAl (b). The main slip system is indicated.

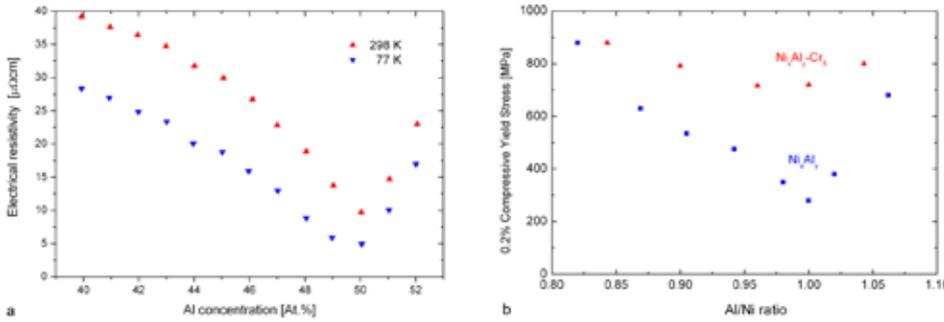
homogenous solid solution phase field around the stoichiometric composition and its congruent melting temperature. Intermetallic NiAl possesses the B2-ordered crystal structure as shown in Fig. 1b. This compound belongs to the realm of Hume Rothery phases [10] stabilized by the valence electron concentration  $e/a = 3:2$ . 'e' is the number of valence electrons and 'a' represents the coexisting atoms in the compound. The asymmetrical solid solution region is characterized by different defect structures - Ni antistructure atoms on Al sites or vacancies in the Ni sublattice as depicted in the schematic drawing of Fig. 2. The optimum thermal stability of the NiAl compound occurs at the stoichiometric composition  $\text{Ni}_{50}\text{Al}_{50}$ . Deviations from the stoichiometry result in the formation of constitutional lattice defects which strongly affect the physical and mechanical properties of NiAl. This is clearly demonstrated by the asymmetry of the electrical resistivity curve (Fig. 3a) [11,12], the hardness or yield stress curves (Fig. 3b) [13], and the lattice parameter as a function of the composition [6]. Generally, vacancies in Al-rich NiAl compounds cause a stronger impact on the physical and mechanical properties than Ni antistructure atoms do [11,14,15]. Therefore, a precise knowledge of the site occupancies of the alloying elements is of great interest regarding improvements in physical and mechanical properties.

An important requirement for a sufficiently ductile and tough material behavior at lower and ambient temperatures is the dislocation mobility on defined crystallographic glide planes and in determined glide directions, as shown in Fig. 1b. The von Mises criterion is fulfilled if five independent slip systems are active in non-ordered fcc, bcc or hcp metals such as Al, Fe, Ni or Co and their alloys, whereas the B2-ordered NiAl compound possesses only three independently operating slip systems, which are:  $\{110\}\langle 111 \rangle$ ,  $\{110\}\langle 110 \rangle$ , and  $\{110\}\langle 100 \rangle$ . The acting



**Fig. 2:** Atomic defect structures of B2-ordered NiAl as a function of the stoichiometry. In Al-rich compositions vacancies are present in the Ni sublattice, whereas in Ni-rich compositions the Al sublattice possesses Ni antistructure atoms.

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**Fig. 3:** Influence of different structural disordering on the electrical resistivity (a) and yield strength (b) as a function of the Al content.

deformation mechanisms are restricted and may not exhibit sufficient ductility and toughness necessary for efficiently operating engine components in energy conversion facilities. However, alloying NiAl with refractory metals reduces the relatively high antiphase boundary energy (APB) of about  $E_{APB} = 850 \pm 40 \text{ mJ/m}^2$  [16,17], which is responsible for a diminished activation of the more sessile  $a\langle 111 \rangle$  superdislocations. Mobile  $a\langle 100 \rangle$  misfit dislocations are formed at the NiAl || Cr(Mo,Re) interfaces [18,19] and these processes combined will increase the ductility of NiAl-based alloys.

Theoretical calculations predict that chromium atoms dissolved in the ordered NiAl lattice exhibit a strong site preference for the Al sublattice of about 70 at % [16,17]. However, a certain amount of Cr atoms are located in the Ni sublattice and diminishes the degree of long range ordering in the B2 lattice which will reduce the APB energy. This prediction was confirmed by high resolution field ion microscopy -APFIM- studies on the atomic scale, where segregations of Cr atoms on APBs were detected [20]. The disturbance of the perfectly ordered NiAl sublattice due to chromium enhances the ductility. This was experimentally verified in uniaxial compression tests and also in four point bend tests performed at room temperature [19].

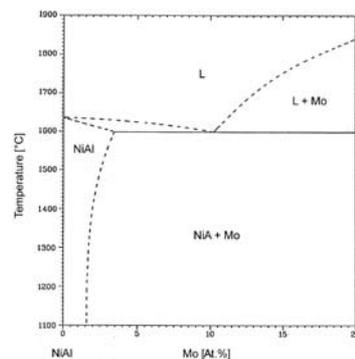
Hardness and strength of binary NiAl or ternary NiAl alloys are mainly determined by deviations from this stoichiometry. This is due to the dominant atomic defects, such as vacancies, anti-structure atoms and site preferences of the alloying elements in the B2-ordered lattice [20,21]. The influence of the stoichiometry and the chromium content of 5 wt % on the mechanical properties at NiAl and NiAlCr is illustrated by the asymmetric curves of the flow stress of pure NiAl and of the hypoeutectic  $\text{Ni}_{47.5\pm X}\text{Al}_{47.5\pm X}\text{Cr}_5$  solid solution alloy as a function of the Al concentration as presented in Fig. 3b. The flow stresses exhibit minimum values at the stoichiometric compositions and increase with increasing off-stoichiometry. On the Ni-rich side of the phase diagram moderate solid solution hardening occurs, whereas on the Al-rich side a pronounced increase in flow stress due to a strong vacancy hardening mechanism is induced. From this behaviour it is concluded that Ni antistructure atoms and structural vacancies are contributing as important mechanisms to the strengthening behavior of NiAl. The principal idea behind this is the elastic distortion

of the B2 lattice due to atomic size effects - par-elastic interactions. The greater flow stress values of about  $\sigma_y = 300 \text{ MPa}$  of the  $\text{Ni}_{47.5}\text{Al}_{47.5}\text{Cr}_5$  alloy around the stoichiometry ( $X_{\text{Al/Ni}} = 1$ ) are caused by the stronger influence of chromium additions on the par-elastic and di-elastic solid solution hardening mechanisms. The latter

one is due to the larger elastic modulus of chromium of  $E_{Cr} = 268 \text{ GPa}$  compared to  $E_{\text{NiAl}} = 179 \text{ GPa}$  of NiAl. Maximum flow stresses of  $\sigma_y = 670 \text{ MPa}$  have been achieved on the Ni-rich side at  $X_{\text{Al/Ni}} = 0.87$ . However, the strongest “solid solution hardening effect” occurs on the Al-rich side due to a pronounced vacancy hardening mechanism.

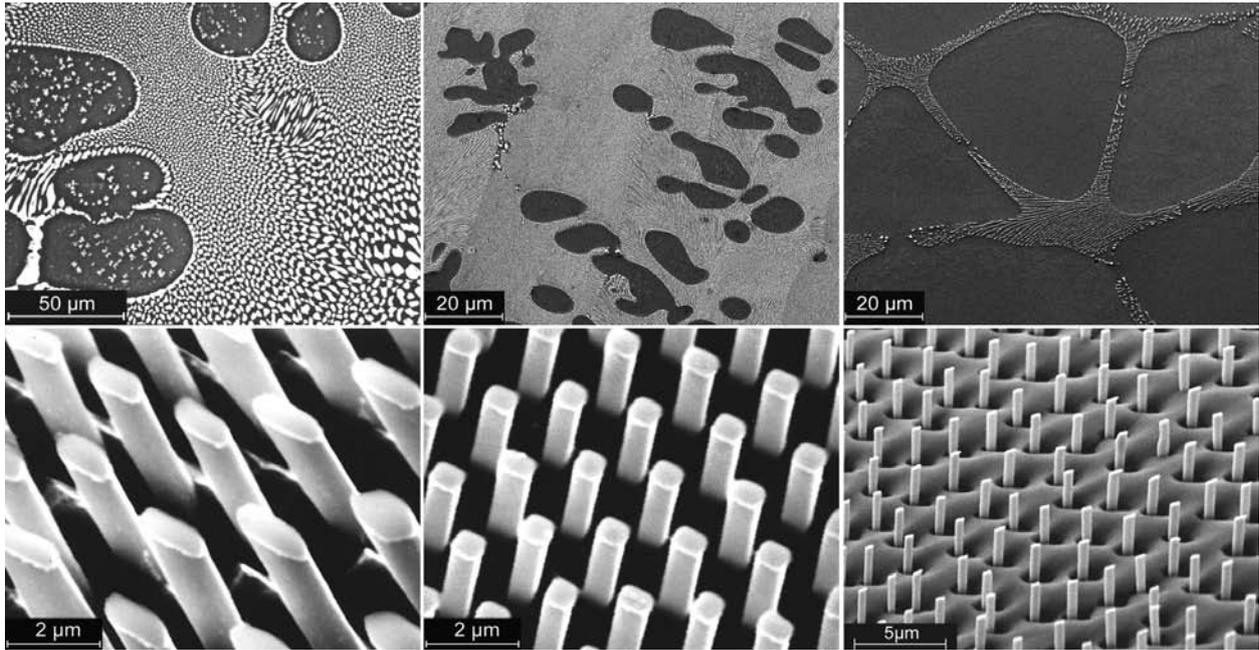
**Reinforcement by High Strength Refractory Metal Fibers.** An appropriate alloy design of NiAl-X materials with improved strength and toughness by macro and micro-alloying is based on the thermodynamic of mixed phases and on the activation of specific deformation and strengthening mechanisms. These are solid solution hardening, fiber reinforcement, and dispersion strengthening which lead to a strong increase in warm strength and creep resistance. The refractory transition metals like vanadium, chromium, molybdenum and tungsten -bcc structure- and the hcp rhenium are forming quasi-binary eutectic systems with NiAl. The directionally solidified fiber-reinforced NiAl-Cr, NiAl-Mo, and NiAl-Re eutectics possess unidirectionally aligned stiff fibres of high melting point and show markedly improved properties, such as high warm strength combined with better ductility in comparison to non-reinforced monolithic NiAl.

The quasi-binary NiAl-X(Cr,Mo,Re) eutectic phase diagrams are representatively illustrated by the NiAl-Mo phase diagram as shown in Fig. 4. The NiAl-rich side exhibits a moderate solubility of about 3.5 at.% Mo at the eutectic temperature of  $T_{eut} = 1600 \text{ }^\circ\text{C}$ . The eutectic concentration was determined to be  $c_{eut} = 9.5 \text{ at.}\%$  [22]. With increasing Mo content the liquidus temperature exhibits a steep increase up to the



**Fig. 4:** Quasi-binary NiAl-Mo phase diagram.

melting temperature of pure molybdenum of  $T_m = 2610 \text{ }^\circ\text{C}$ . It should be mentioned that the solubility of Ni and Al in Mo is rather low of the order of  $c_{sol} < 0.5 \text{ at.}\%$  at the eutectic temperature. The quasi-binary NiAl-Cr and NiAl-Re phase diagrams exhibit different eutectic compositions of



**Fig. 5:** SEM-images showing typical microstructures of hypo-eutectic NiAl-Cr<sub>27</sub> (a), NiAl-Mo<sub>8</sub> (b), and NiAl-Re<sub>1</sub> (c) alloys in the as-cast condition as well as of unidirectionally solidified NiAl-Cr<sub>34</sub> (d), NiAl-Mo<sub>9.5</sub> (e), and NiAl-Re<sub>1.25</sub> eutectics (f).

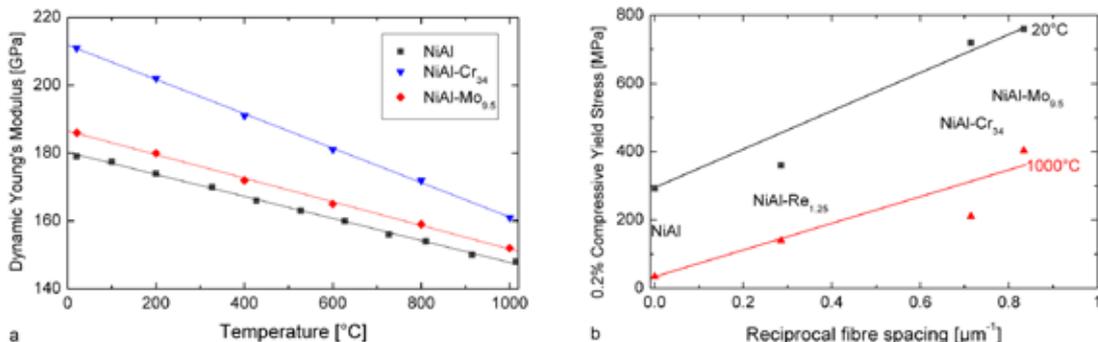
$c_{eut} = 34$  at.% Cr and  $c_{eut} = 1.25$  at.% Re. The related eutectic temperatures are  $T_{eut} = 1450$  °C and  $T_{eut} = 1668$  °C [8,23] and the melting temperatures of the refractory metals Cr and Re are  $T_m = 1880$  °C and  $T_m = 3180$  °C [24], respectively. The NiAl-Re eutectic system is of great scientific and technological interest due to its extraordinary high eutectic melting temperature which results in very thermally stable microstructures of this composite.

**Microstructures and Mechanical Properties of NiAl-X(Cr,Mo,Re) Alloys.** The hypoeutectic NiAl-X(Cr,Mo,Re) alloys exhibit in the as-cast condition similar microstructural features as shown in Figs. 5a to c. These consist of primary NiAl crystals containing finely dispersed Cr, Mo, or Re precipitations embedded in the fibrous eutectics with thin Cr, Mo, or Re fibers of 0.2 to 1.2  $\mu\text{m}$  in thickness. The microstructures of the unidirectionally solidified eutectics, presented in Figs. 5d to f, reveal periodic arrangements of the Cr, Mo, or Re fibers of elliptical (NiAl-Cr), rectangular (NiAl-Mo), or square-shaped (NiAl-Re) cross sections. The average diameter or edge length of the fiber cross sections ranges from 0.3 to 1.8  $\mu\text{m}$ .

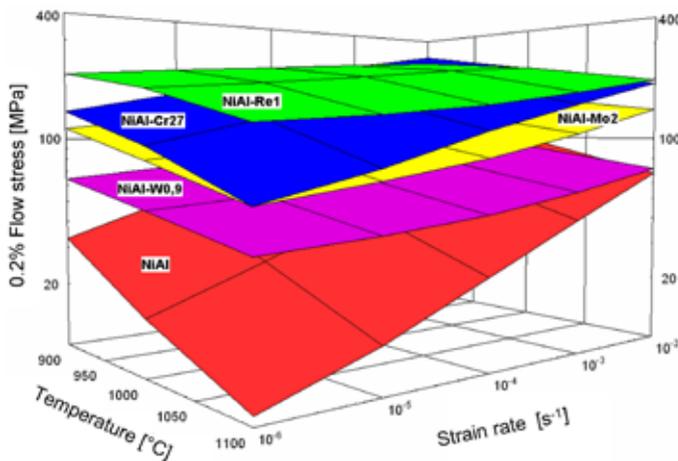
Elasticity is an important material property for the constructional layout of engine components. The elastic moduli of the NiAl intermetallic compound are basically determined by its relatively high lattice energy of 470 kJ/mole [25] and interatomic bond length (NiAl) atoms of  $r_{Ni+Al} = 249.7$  pm in the B2 lattice. The Young's moduli of the unidirectionally solidified NiAl-X(Cr,Mo) eutectics are increased due to the high elastic stiffness of the reinforcing fibres of the refractory transition metals Cr, Mo, and Re by taking their volume fractions into account according to the model of the parallel array - iso-strain condition - described by the equation:

$$\bar{E}_{eut} = E_{NiAl} \cdot V_{NiAl} + E_X \cdot (1 - V_{NiAl})$$

The temperature dependence of the Young's moduli of the stoichiometric single phase NiAl and of the NiAl-Cr<sub>34</sub> and NiAl-Mo<sub>9.5</sub> eutectics are illustrated in Fig. 6a. NiAl-Cr<sub>34</sub> exhibits a maximum elastic modulus of about 210 GPa at room temperature. At 1000 °C the Young's modulus is still quite high of the order of 160 GPa, whereas the NiAl-Mo<sub>9.5</sub> eutectic shows  $E \approx 150$  GPa, respectively [15]. The elastic moduli of pure NiAl and NiAl-Re<sub>1.25</sub> are almost the same. This is



**Fig. 6:** Young's moduli (a) and yield strengths (b) of the unidirectionally solidified NiAl-Cr<sub>34</sub>, NiAl-Mo<sub>9.5</sub>, and NiAl-Re<sub>1.25</sub> eutectics as a function of temperature and reciprocal fiber spacing.



**Fig. 7:** High temperature strength of hypo-eutectic NiAl-X(Cr,Mo,Re,W) alloys in the as-cast condition as a function of temperature and strain rate in spatial representation.

due to the small volume fraction of discontinuous Re fibers present in the eutectic microstructure. It should be mentioned that the specific moduli ( $E/\rho$ ) of NiAl and NiAl-based alloys are quite high of  $E/\rho(\text{NiAl-Cr}) = 33 \text{ GPa/gcm}^{-3}$ , compared to that of nickel or cobalt base superalloys, showing  $E/\rho \approx 25 \text{ GPa/gcm}^{-3}$ .

The compressive yield stresses of the unidirectionally solidified NiAl-X(Cr, Mo, Re) eutectics and of a hot extruded  $50 \mu\text{m}$  grain size NiAl [19] are plotted in Fig. 6b as a function of the reciprocal fiber spacing  $d_f^{-1}$  for two different temperatures of  $T_1 = 20 \text{ }^\circ\text{C}$  and  $T_2 = 1000 \text{ }^\circ\text{C}$ . The strong increase in the flow stress with decreasing fibre spacings are quantitatively described by the expression:

$$\sigma_y^c = \sigma_y^{\text{NiAl}} + k d_f^{-1}$$

$\sigma_y^{\text{NiAl}}$  is the yield stress of NiAl,  $k = \bar{M} \cdot G^{\text{NiAl}} \cdot |\vec{b}|$ ,  $G^{\text{NiAl}}$  is the shear modulus,  $\vec{b}$  is the Burgers' vector, and  $\bar{M}$  is an average Taylor factor.

The governing high temperature deformation and strengthening mechanism, such as dislocation creep and dispersion hardening at low strain rates - creep behavior -, of NiAl are similar to those of non-ordered metals and alloys despite the fact the NiAl matrix is ordered even at high temperatures up to the melting point [26]. The topographic map in Fig. 7 illustrates the spatial representation of the stress as a function of the test temperature and strain rate. Monophase NiAl softens dramatically and exhibits the lowest creep stress. Alloying NiAl with the bcc refractory metals W, Mo and Cr increases remarkably the warm strength at low strain rates. However, NiAl-Re<sub>1</sub> reveals optimum high temperature strength of  $\geq 150 \text{ MPa}$  under quasi creep conditions.

**Application.** In joint projects with partners of the automotive and power generation industries, the MPIE took part in the development and characterization of these advanced high temperature materials. Various structural components - injection pins for fuel injections - of internal combustion engines, - precision thermal tiles for linings -, in combustion chambers of stationary gas turbines of power plants, and - static blades - in jet engines have been produced by die casting and metal powder injection molding

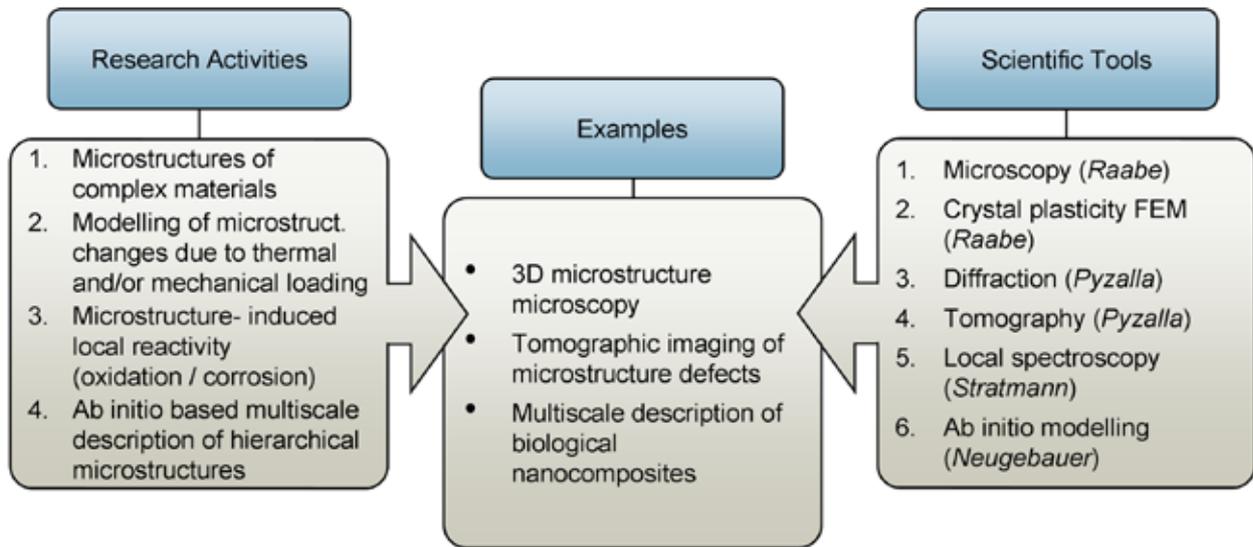
(MIM). Operational reliable component testing procedures have been successfully performed and the results were evaluated with respect to improved engine efficiency.

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# Microstructure-Related Materials Properties



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## 2- and 3-Dimensional Orientation Microscopy Investigations on the Nucleation Mechanisms of the Cube Recrystallisation Texture in a Cold Rolled Fe-36%Ni Alloy

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The present paper presents new results on the nucleation mechanism leading to the formation of the sharp cube texture during annealing of heavily cold rolled fcc metals with high stacking fault energy. It has been proposed in literature that the stored energy difference between areas of cube orientation and those of other texture components is the reason for the preferred nucleation of cube grains. Using a new technique for stored energy measurements we were able to show that this is most probably not correct because the stored energy differences are actually rather small. Using 3D orientation microscopy we also show that the grain boundary character does not play a significant role for cube nucleation either. Rather, it is the high mobility of the dislocation arrangement in cube grains which leads to their quick recovery in the first stage of annealing.

The mechanisms for the formation of the sharp cube texture ( $\{001\}\langle 100 \rangle$  preferred orientations) during recrystallisation of heavily cold rolled metals with fcc structure and high stacking fault energy have been subject of intense research and dispute for more than 60 years. Nowadays, a general agreement has been achieved that oriented nucleation in pre-existing cube bands is the main reason for the cube texture formation (see, for example [1-7]).

For aluminium it has been shown that the cube nucleation occurs from deformed cube bands by grain boundary bulging [12]. The same has been observed for copper by Duggan et al. [5] although the authors of this paper called the nucleation mechanism "micro-oriented growth" at that time. Understanding the details of the nucleus formation mechanism is difficult for aluminium and copper because they recover already at room temperature but even more rapidly at elevated temperatures occurring during the cold rolling process. Fe-36%Ni, in contrast is an fcc alloy where the high alloying content leads to high dislocation glide resistance and the high melting point to a high resistance against dislocation climb. Consequently, this alloy really shows the frozen-in deformation structure after cold rolling. The alloy is therefore an ideal candidate to study nucleation of the cube texture in more details.

Earlier work on this alloy [11] suggested that a nucleation mechanism based on abnormal subgrain growth of cube cells [9] would be responsible for the formation of cube oriented grains. Julliard et al. [13]

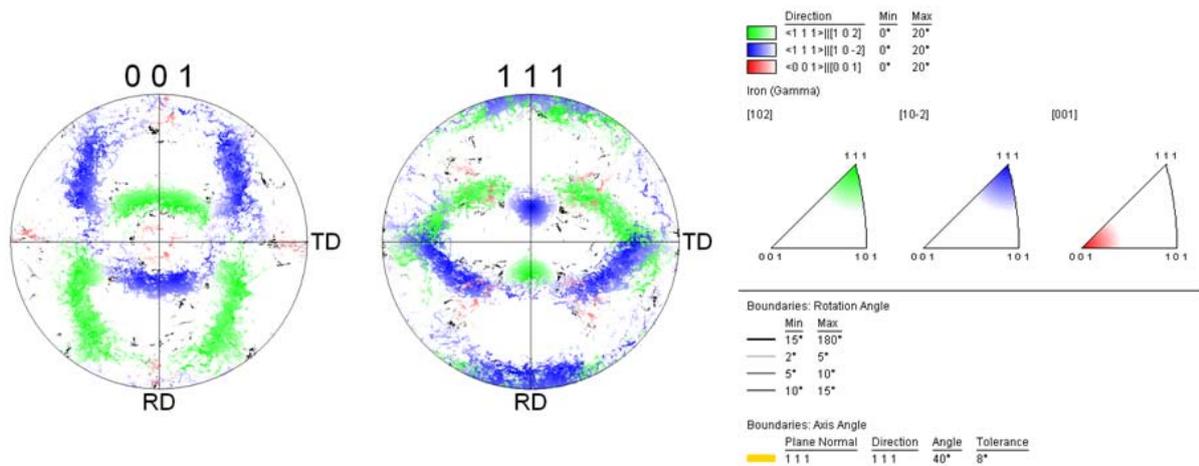
showed, in contrast, that the material nucleates by grain boundary bulging from the deformed cube bands and claimed that the stored energy difference between cube-oriented grains and others is the reason for the bulging [10]. Here we present a number of new investigations on the subject using high resolution 2D and 3D EBSD-based orientation microscopy (EBSD: electron backscatter diffraction). They show, in particular, that the stored energy distribution after cold rolling is most probably not the first reason for the formation of the cube texture.

**Experimental details.** Cold rolled material with a deformation degree of 94 % was supplied by Imphy S.A.. Recrystallisation experiments were carried out on this material by introducing samples into a preheated furnace at 600 °C. Short annealing times of 1 and 2 minutes allowed observation of the nucleation process.

High resolution EBSD-based orientation microscopy was carried out using a JEOL 6500 F SEM and a TSL/EDAX EBSD system. The EBSD pattern analysis was performed in a high angular resolution mode. Measurement step sizes between 30 and 50 nm were selected which are close to the physical lateral resolution of the technique.

In order to observe the 3-dimensional (3D) microstructure of the growing nuclei 3D orientation microscopy on the basis of FIB-EBSD tomography was applied as well (FIB: focused ion beam microscope). The measurements were carried out on our self-developed system described in [14] on a Zeiss Crossbeam 1530 XB FIB-SEM. A low resolution Hough transform was selected. The measurement step size was  $100 \times 100 \times 100$  nm and volumes of approx.  $20 \times 20 \times 10 \mu\text{m}^3$  were measured.

**Cold rolled material: Microstructure and stored energy distribution.** Fig. 1 (next page) shows the texture of the cold rolled material obtained from one large EBSD scan in form of pole figures. At the same time this figure serves as colour legend for all subsequent scans: 3 colour shades are used to describe the most important texture fibres. Shades of green and blue describe the two fibres  $\langle 111 \rangle_{\text{crystal}} \parallel [102]_{\text{sample}}$  and  $\langle 111 \rangle_{\text{crystal}} \parallel [102]_{\text{sample}}$  which comprise all typical cold rolling texture components, Brass  $\{011\}\langle 211 \rangle$ , Goss  $\{011\}\langle 100 \rangle$ , S  $\{123\}\langle 634 \rangle$  and C  $\{112\}\langle 111 \rangle$ . Shades of red

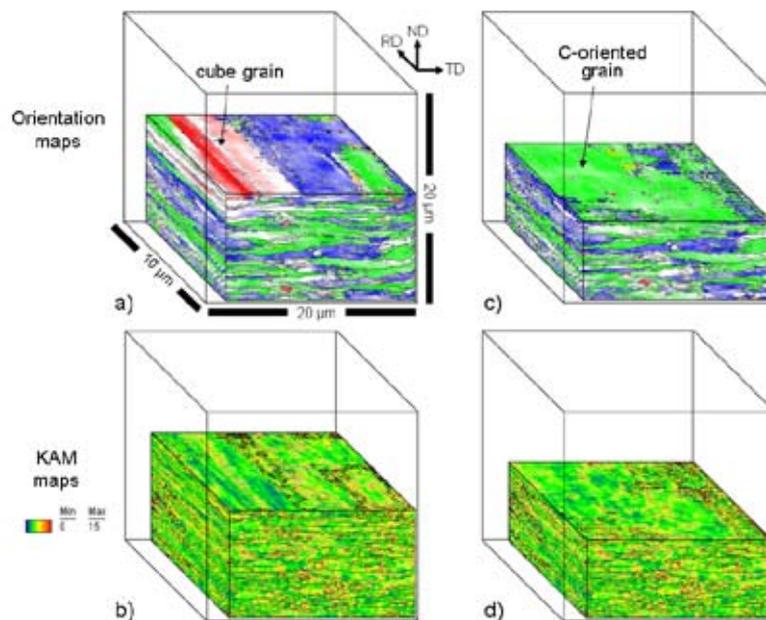


**Fig. 1:** EBSD-measured  $\{001\}$  and  $\{111\}$  pole figures of the cold rolled sample. The colouring scheme is described further in the text. The figure serves as a legend for all following orientation maps.

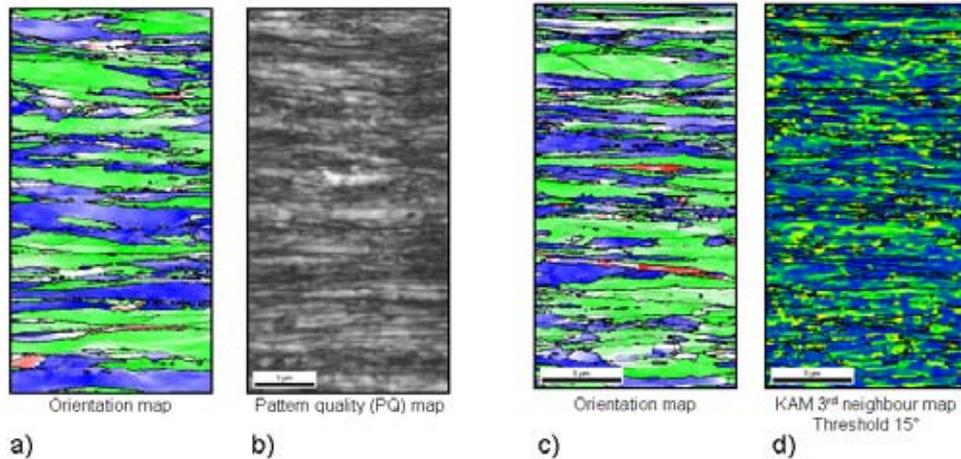
show the cube component  $\{001\}\langle 100 \rangle$ . Fig. 2a shows an example of a cube-oriented band in the cold rolled microstructure observed by 3D orientation microscopy. All morphological and orientational features observed in these maps are in agreement with earlier TEM observations ([11]) with the important exception that the TEM observations clearly showed a well developed cell structure in the cube oriented areas which was not observed in the EBSD-based maps. This observation is an important hint on the nucleation mechanism: they show that the dislocations in cube oriented areas are rather mobile, leading to quick recovery during thin-foil preparation for TEM.

An important parameter for understanding the deformation and recrystallisation behaviour of a material is the deformation energy stored in form

of lattice defects. The determination of orientation resolved stored energy has been classically carried out by means of X-ray or neutron diffraction peak broadening measurements. These techniques, however, suffer from rather complex assumptions and not in all cases satisfying theory. Alternative methods for stored energy measurements are provided by EBSD-based orientation microscopy. One method that has been proposed in literature uses the diffraction pattern quality (PQ) as an indirect measure of dislocation densities and thus stored energy. Recently the present author introduced another EBSD-based method [15], which is based on the interpretation of lattice rotations in terms of geometrically necessary dislocations, GND. The local lattice rotations are evaluated on the basis of the kernel average misorientation (KAM) function. From the KAM values a lower bound for the stored energy



**Fig. 2:** 3D microstructure maps of the cold rolled material measured by 3D EBSD-based orientation microscopy. The top figures a), c) indicate crystal orientations, the bottom figures b), d) show  $1^\circ$ -neighbour KAM maps. The left side figures show a cube-oriented area, the right side a C-oriented one. Both show almost similarly low KAM values, i.e. low stored energy.



**Fig. 3:** Measurements for stored energy determination. a) EBSD orientation map and b) pattern quality map of the cold rolled material. c) EBSD orientation map and d) 3<sup>rd</sup> order KAM map of the cold rolled material. Only misorientations up to 15° are considered in the KAM calculation.

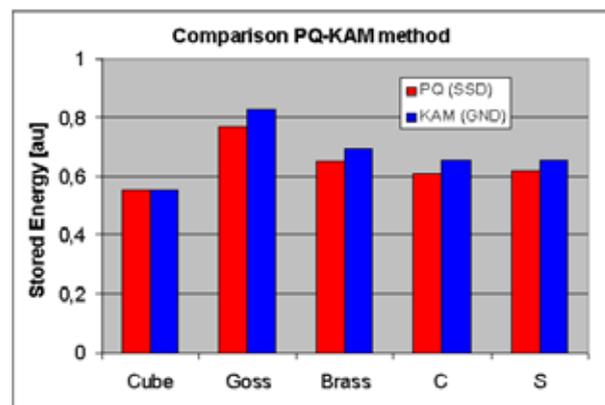
is calculated using the Read-Shockley equation (this equation assumes that the dislocations that cause the lattice rotation are organized in form of low angle tilt boundaries).

We applied the PQ and the KAM method to determine the stored energy of the cold rolled material (see [15] for more details). A first impression of the stored energy distribution is given by Fig. 2b which displays a KAM map of the same area as figure a). Clearly, the cube oriented area shows lower values than its direct surrounding, though the differences are small. On the other hand also other grains show similarly low KAM values as displayed in Fig. 2c and d. For a more quantitative evaluation high resolution 2D maps were measured as well. Fig. 3 shows two orientation maps and the respective PQ and KAM maps. From the maps average stored energy data were determined for a number of different texture components, namely Cube, Goss, Brass, S and C. The results are displayed in Fig. 4 in form of the stored energies of the different texture components, normalized to that of the cube texture. The surprising result of this classification is that the stored energy varies only little for the different texture components and that the cube component does not have a particularly low energy although it has the lowest stored energy of all components. Stored energy differences in the cold rolled material can therefore most probably be excluded as the fundamental reason for the selection of the cube orientation during recrystallisation.

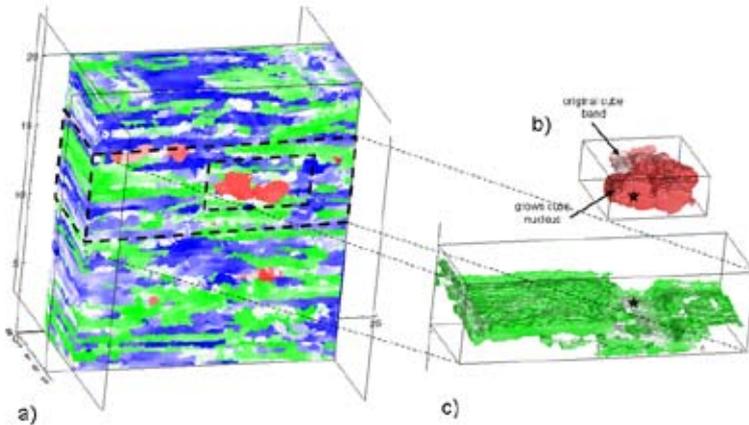
**Shortly annealed material: Grain boundary character of nuclei.** Fig. 5 shows the 3D microstructure of the sample annealed for 1 minute. It is visible, that parts of the cube band still show the same structure as the deformed material. However, some areas have bulged out and grown out of the cube band. From the residual cube band neighbourhood it is possible to determine the original surrounding of cube grains

before they started to bulge out. The misorientations between the point around that marked in the 3D microstructure map in Fig. 5c and the orientation of the growing cube grain displayed in Fig. 5b were measured and found to be all in the order of a 51° rotation about an approximate  $\langle 3\ 6\ 7 \rangle$  axis. Thus, the nucleus, when it started to grow, did not have a special boundary, for example of a  $38^\circ \langle 111 \rangle (\Sigma 7)$  type. It can therefore be concluded that the micro-oriented growth assumption as proposed for example by Duggan et al. [5] can be excluded as a possible cube orientation selection mechanisms.

**Considerations on the recovery process.** Having excluded stored energy and special boundaries as possible reasons for the cube orientation selection there is one explanation for the cube texture formation remaining which is a superior recovery rate of cube oriented areas. As proposed by Ridha and Hutchinson [8] the reason for this high recovery rate may be the particularly mobile dislocation arrangement that is formed during the deformation of cube grains. In the present work recovery of cube grains has been shown to be much quicker than



**Fig. 4:** Relative stored energies of different texture components measured with the PQ and the KAM method on cold deformed material.



**Fig. 5:** 3D microstructure map of a sample annealed for 1 minute. a) shows the total material block; b) a growing cube grain which is still connected to the original cube band from which it grew out. c) displays the direct neighbourhood of the growing cube grain. Points that have been used for misorientation determination between the growing nucleus and its original neighbourhood (see text) are indicated by asterisks.

that of other orientations: while the stored energy in cube-oriented areas drastically drops in the early stage of annealing, all other orientations mainly keep their deformation structure over much longer annealing times.

Using the KAM method for the quantitative determination of stored energy and literature values for the energy of boundaries it is possible to calculate the critical bulging radius for nucleation (see [15] for more details). In the present case a critical radius of  $0.74 \mu\text{m}$  was calculated which is about twice the value observed by TEM [11]. The real bulging radius would require – according to our calculations – a stored energy about twice as large as the one calculated by KAM. The reason for this discrepancy is two-fold: first, the KAM calculations only consider the geometrically necessary dislocations and disregards the statistically stored ones. Second, the KAM analysis using a Read-Shockley approach displays only a lower bound for the stored energy as it actually assumes the dislocations to arrange in the configuration with lowest possible energy. It seems, nevertheless, that the KAM calculations are a good approximation of the stored energy, at least for the present material system.

**Conclusions.** Nucleation of cube grains occurs by grain boundary bulging of cube bands after effective recovery. Possible reasons for the preferred bulging of cube grains have been carefully checked:

- (i) The 3D orientation microscopy data allow excluding any influence of special grain boundaries, in particular of  $\Sigma 7$  ( $38^\circ \langle 111 \rangle$ ) boundaries on the nucleation event. No  $\Sigma 7$  boundaries were found at the origin of successfully grown nuclei.
- (ii) The stored energy differences between cube and non-cube areas in the cold rolled material have been shown to be rather small

( $E_{\text{cube}} \approx 0.8 E_{\text{non cube}}$ ). These energy differences are not sufficient to explain the formation of the sharp cube texture. However, after a short annealing the stored energy differences increased to  $E_{\text{cube}} \approx 0.6 E_{\text{non cube}}$ .

(iii) Exclusion of points (i) and (ii) leaves only one possible explanation, the high recovery rate of the dislocation structure formed during deformation of cube grains in accordance with ideas proposed already in 1982 by Ridha and Hutchinson [8]. The high recovery rate leads to rapid cell growth and a significant energy difference to the surrounding grains which then promotes rapid growth after recovery. Before annealing the absolute value of the stored energy difference was found to be too large to

allow bulging of a large angle grain boundary. After a short annealing this difference drastically increased due to recovery and may then lead to successful bulging.

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# Soft Magnetic Iron-Silicon and Iron–Silicon–Aluminium Alloys for Industrial Applications Processed under Microgravity

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More than 1 million tons of grain-oriented iron–silicon sheet are produced each year in the worldwide steel industry, and the improvement of this technologically important material and its production methods are still an ongoing research process [1,2]. Due to their excellent soft magnetic properties, these electrical steels are used for electric motors, transformers, generators and static induction devices. An ideal soft magnetic material should have a high magnetic permeability and induction and at the same time the core losses produced during cyclic magnetization processes must be as low as possible. The financial losses due to the power losses which result from using material with non-optimized magnetic properties in German transformer stations amount to several hundred million Euros per year. Today's industrially used iron–silicon alloys have a silicon content of about 3 wt%. Optimal magnetic properties and minimal core losses, however, can be achieved when employing alloys with higher silicon contents in the range of about 5.5 to 6.5 wt%. In this composition range, the permeability reaches a maximum, the coercivity is minimal, and the magnetostriction decreases to zero resulting in noise-free power transmission.

Unfortunately, such improvement of the magnetic properties is accompanied by a dramatic decrease in ductility at about 4 to 5 wt.% Si making conventional processing and specifically the required cold rolling of such alloys almost impossible. The causes for such deterioration of the mechanical properties are generally known. Besides silicon segregation processes during solidification (see e.g. [3]), the decisive factor is the formation of B2 and D0<sub>3</sub> superlattices with increasing silicon content [4] (see Fig. 1). The influence of atomic order on the electrical and magnetic properties is still an active area of research [5,6]. A possible approach to suppress the ordering tendency is a partial replacement of silicon by aluminium [7]. Although the effect of aluminium on the soft magnetic properties is not as beneficial as that of silicon, the important advantage is that ordering of the aluminium atoms occurs only at a much higher solute atom content of 10 wt.% than in case of silicon.

Binary Fe–Si alloys with 3.5 to 6.5 wt.% Si and ternary Fe–Si–Al alloys with 4.5 to 6.5 wt.% (Si+Al) were molten and solidified under microgravity conditions in a levitating state during parabolic flight experiments. The resulting microstructures were analysed by LOM, SEM, TEM, and XRD as well as hardness measurements in order to find out if segregation to grain boundaries is affected

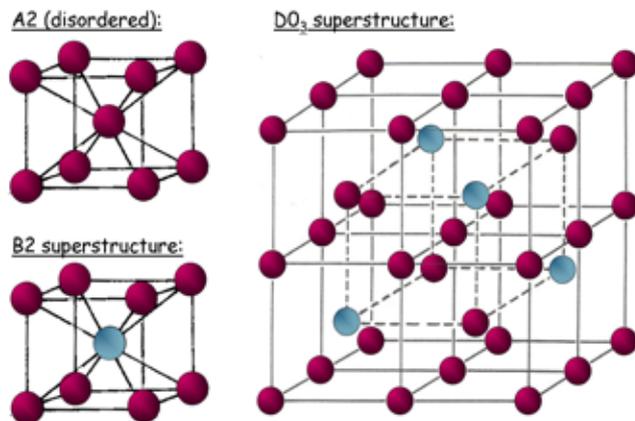
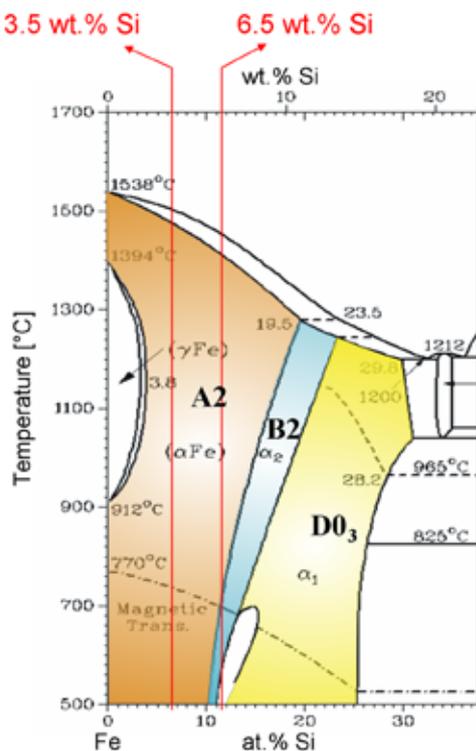


Fig. 1: Fe-rich part of the Fe-Si binary phase diagram (data taken from [16]) and sketches of the disordered Fe(Si) bcc lattice and the ordered B2 and D0<sub>3</sub> superstructures.



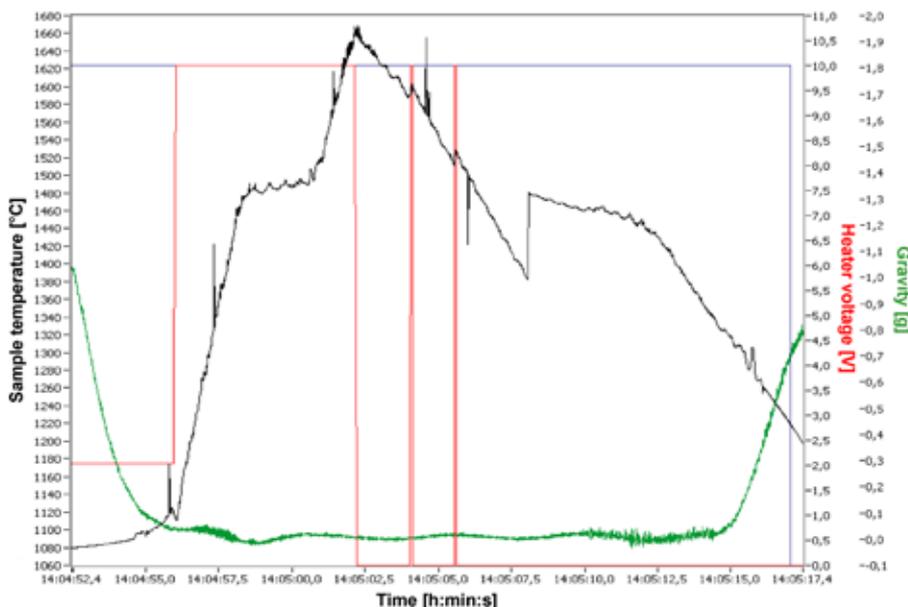
**Fig. 2:** Machined sphere of the as-cast Fe-3.5 wt.% Si alloy after etching (65% H<sub>2</sub>O, 30% HNO<sub>3</sub>, and 5% HF). The diameter is 6 mm.

by microgravity and if ordering tendencies can be suppressed. Additionally, the viscosity and surface tension of the melt was determined from the oscillating drop.

From induction-melted ingots of the binary and ternary Fe–Si(–Al) alloys, spheres of 6 mm in diameter were machined (Fig. 2). For the parabolic flight experiments, the spheres are inserted into the so-called TEMPUS facility which is a special electromagnetic levitation device that has been developed at the DLR (German Aerospace Center) for experiments under microgravity [8]. The acronym

TEMPUS is the German abbreviation for "crucible-free electromagnetic positioning in zero gravity". A typical time-temperature plot of a parabolic flight experiment is shown in Fig. 3 for a Fe-3.5 wt.% Si sample. As the green curve shows, microgravity conditions exist for about 20 seconds. After the onset of microgravity, the levitating, pre-heated Fe–Si sphere is quickly heated up from 1080°C above its liquidus temperature and cooled down again. The first plateau in the heating curve at temperatures between about 1480 and 1500°C indicates the melting interval of the sample. The liquid sample is heated to about 160°C above its melting point and then the heater is switched off. During cooling, forced oscillations of the liquid drop are brought about by two

short heating pulses (red line). From the frequency and damping of these oscillations, surface tension and viscosity of the melt can be determined [9,10,11]. Before solidification, liquid samples often show the effect of undercooling due to missing nucleation sites. If undercooling is unwanted, nucleation can be triggered in the parabolic flight experiments by contacting the liquid drop with a needle causing heterogeneous nucleation. In the experiment described in Fig. 3, the sample undercooled freely to about 100°C below its solidus temperature. The onset of nucleation is marked by a strong



**Fig. 3:** Sample temperature (black), gravity (green), and heater power (red) as a function of time during a parabolic flight experiment processing a Fe-3.5 wt.% Si sphere in the TEMPUS facility.



recalescence effect. Due to the liberation of the latent heat of crystallization, the sample is rapidly heated up again before finally cooling to room temperature. Microgravity conditions are maintained until solidification is completed.

From the evaluation of forced oscillation data from different parabolas, the surface tension  $\sigma$  can be determined as a function of temperature. In order to compare the different binary and ternary Fe-Si(-Al) alloys, Fig. 4 shows the surface tension at the liquidus temperature  $\sigma_L$  in dependence on composition. The value  $\sigma_L = 1.92$  N/m for the surface tension of pure iron is taken from Brillo and Egry [12] and was determined by the same oscillating drop technique. Earlier values for pure iron measured with the sessile drop method are somewhat lower and vary in the range 1.72 to 1.87 N/m [13,14]. The present data indicate that the addition of silicon slightly reduces the surface tension. The replacement of silicon by aluminium results in a further reduction of the values. The viscosity  $\eta$  of the melt is calculated from the damping behaviour of the forced oscillations of the liquid drop. As the resulting values have only low accuracy and scatter in a broad range between 10 and 25 mPa·s with estimated errors reaching  $\pm 5$  mPa·s, no clear conclusion can be drawn on the effect of silicon and aluminium additions. The recommended IUPAC value for the viscosity of pure iron at its melting point is significantly lower and amounts to  $\eta_L = 5.9 \pm 0.4$  mPa·s [15].

Inspection of cross sections of the solidified spheres by optical and electron microscopy as well as EDX- and hardness scans indicate that all samples were chemically homogenous, single-phase material. Large cavities were found in the central

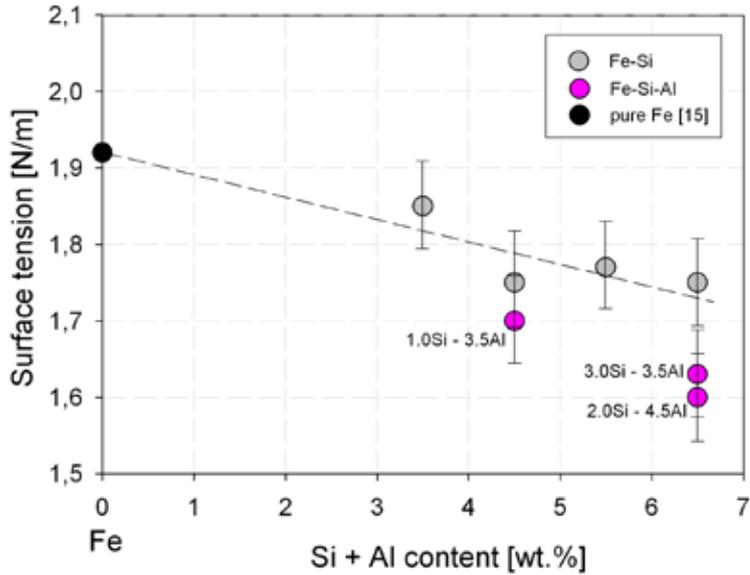


Fig. 4: Surface tension of Fe-Si and Fe-Si-Al alloys as obtained from analysis of forced oscillations of the levitating liquid drop in the TEMPUS facility under microgravity conditions in a parabolic flight experiment.

part of the spheres as a result of the solidification process proceeding from the outer scale to the core. The microstructure consists of large grains of several hundred  $\mu\text{m}$  in diameter with some subgrain structure visible. Composition and hardness is the same as in the as-cast state before the parabolic flight experiments. Silicon as well as aluminium have a strong solid solution hardening effect. However, the hardening effect of aluminium is only about half of that of silicon as is shown in Fig. 5.

In SEM-EDX scans across grain boundaries, no segregation of silicon or aluminium to the grain boundaries is visible. Respective TEM-EDX measurements confirm this result. A central point of interest is the occurrence of superstructures, i.e. ordering of the silicon or aluminium atoms on the bcc Fe lattice. In case of the binary Fe-Si alloys, TEM

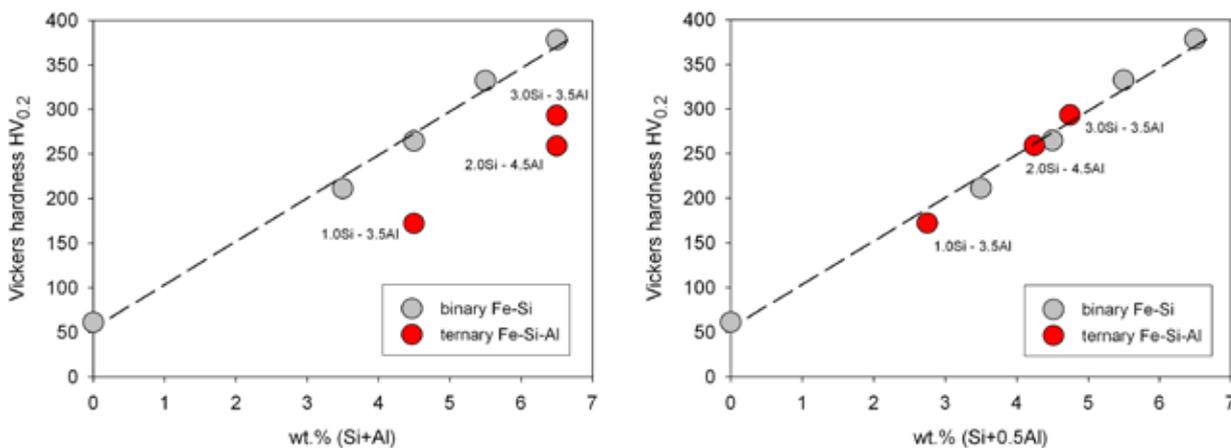


Fig. 5: Vickers hardness  $HV_{0.2}$  of Fe-Si and Fe-Si-Al alloys showing a strong solid solution hardening effect of Si and Al. A plot of the hardness as a function of the weighted alloying content  $\text{Si}+0.5\text{Al}$  (right side) shows that the hardening effect of Si is about twice as strong as that of Al.



selected area electron diffraction patterns reveal the occurrence of ordered B2 and  $DO_3$  superstructure reflections in accordance with the phase diagram. Whereas in an alloy with 3.5 wt.% Si, there is no indication of ordering, the alloy with 5.5 wt.% Si shows some weak superstructure reflections which are clearly visible for the alloy with 6.5 wt.% Si. However, in case of the ternary alloys XRD gives no indication of any ordering effect up to 6.5 wt.% Si+Al, and in the TEM electron diffraction patterns of respective alloys, superstructure reflections of B2 or  $DO_3$  ordering are only hardly detectable even in alloys with 6.5 wt.% Si+Al. Obviously, the replacement of silicon by aluminium results in 'nearly completely' disordered alloys up to alloying content of 6.5 wt.%. Therefore, Fe-Si-Al alloys of the composition Fe-(1.5-3)wt.%Si-(1-3)wt.%Al are recommended as electric steel grades for transformer sheet applications with minimum core losses.

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## Mechanisms of Internal Stress Formation in Iron Oxide Scales

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The formation of oxide scales on metallic substrates is invariably accompanied by growth incompatibilities. These mismatches give rise to internal stresses capable of deteriorating the shielding efficiency of those surface layers against the destructive reaction between the metal and the corrosive agents [1].

The sources of growth stresses within oxide scales are manifold. Among the diverse models proposed over the past years [2], volumetric strains between the oxide formed and the metal consumed [3], the growth of new oxide within oxide grain boundaries [4-6], phase transformations and epitaxial growth [7,8] appear to be the most relevant mechanisms.

In addition to growth strains, the crystallographic orientation of the substrate can also influence the oxidation kinetics [9-12]. The distinct oxidation behavior of single crystals might be associated therefore with particular internal stress states [13] resulting from preferential orientations of oxide growth. However, internal stresses have been mostly studied in oxide scales forming on polycrystalline substrates [14-17]. Under these conditions, the role of substrate orientation in the crystallographic texture of the oxides and therefore in the internal stresses of the oxide scale can not be evidenced.

The present work was therefore devoted to the influence of substrate orientation and preferential orientation of oxide growth on the internal stress state of multi-phase oxide scales [18-21]. To this end, the evolution of microstructure and internal stresses in oxide scales growing within the high- (650°C) and low-temperature (450°C) oxidation regimes of  $\alpha$ -iron was studied on polycrystals and single crystals of pure iron by in-situ energy-dispersive (ED) X-ray diffraction (XRD) at the Materials Science Beamline EDDI of the Helmholtz Zentrum Berlin at the synchrotron storage ring BESSY and by ex-situ electron backscatter diffraction (EBSD). In contrast to time-consuming angle-dispersive diffraction experiments, EDXRD provides complete diffractograms for fixed sample and detector positions. This reduces considerably the data acquisition times [22,23], enabling therefore simultaneous texture and stress studies in oxide scales. With respect to the lack of information on the gradients of growth stresses during oxidation, stress

analyses by ED diffraction further benefit from the different energies  $E_{hkl}$  of the individual diffraction lines. Thus, each reflection can be assigned to a different information depth. This allows for in-situ depth-resolved stress studies in the near-surface zone [24]. The connection between the microstructure and the internal stress state of the oxide scales was provided by complementary scanning electron microscopy (SEM) and EBSD investigations.

The results of the in-situ phase analyses of iron oxide scales growing on iron polycrystals and single crystals show that oxidation at 450 °C simultaneously leads to the spontaneous formation of magnetite ( $\text{Fe}_3\text{O}_4$ ) and hematite ( $\text{Fe}_2\text{O}_3$ ) on the iron substrates. After 100min of oxidation a partial transformation of magnetite to hematite is caused by an increasing coalescence of voids at the scale interface with the substrate. Above 570 °C wüstite ( $\text{FeO}$ ), the third iron oxide phase with a highly defective crystal lattice, becomes stable. Oxidation at 650 °C (Fig. 1), thus, causes wüstite to rapidly grow predominantly by iron cation diffusion, followed by the formation of thin layers of magnetite and hematite on top of the scale. The type of substrate (polycrystal or different single crystal orientations) does not have an appreciable effect on the growth rate of rapidly growing iron oxide scales at 650 °C, whereas at 450 °C polycrystals oxidize faster than single crystals during the first hour of oxidation due to the presence of grain boundaries on the oxidizing surface.

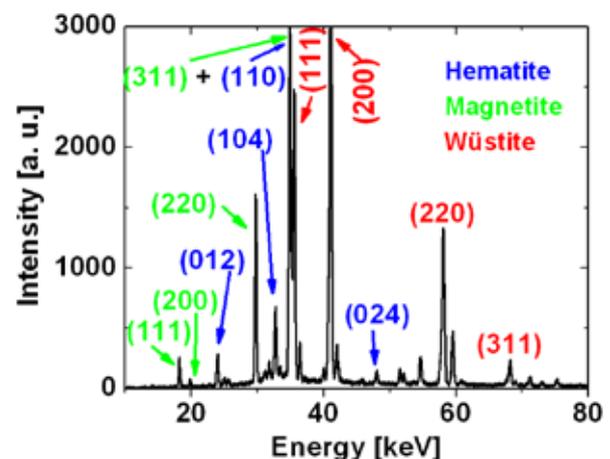
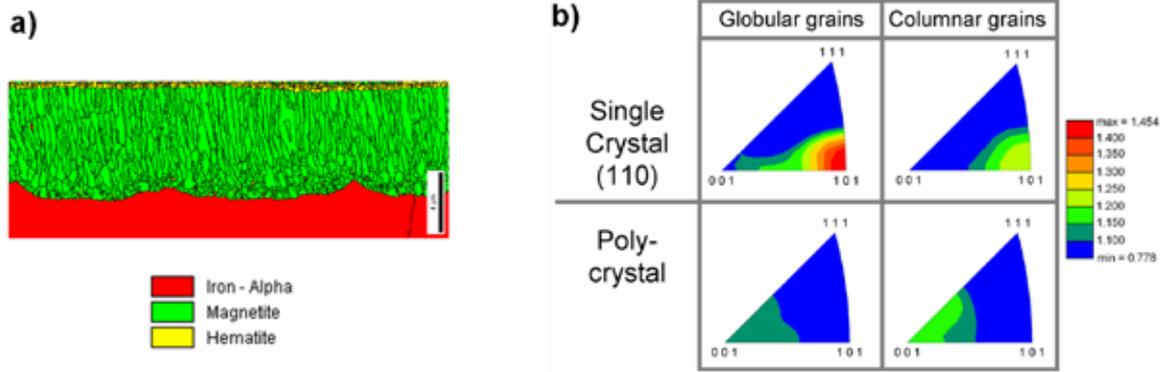


Fig.1: ED-diffractogram obtained during iron oxidation at 650 °C at EDDI.

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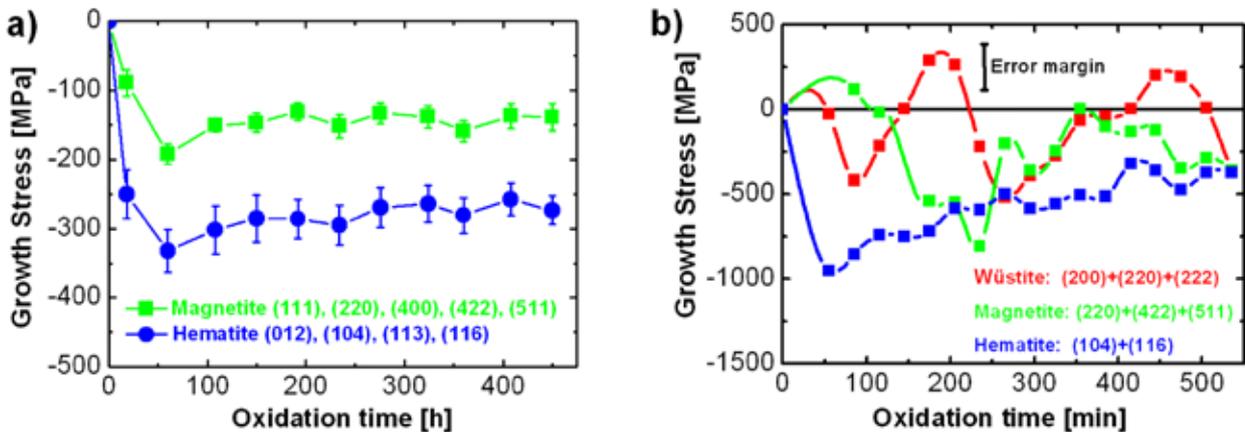
**Fig.2:** (a) EBSD of iron oxide scale grown on an iron polycrystal at 450°C for 5h; (b) Inverse pole figures of magnetite obtained by EBSD after 1h of oxidation at 450 °C.

The microstructure of iron oxide layers is generally characterized by a columnar grain growth of the oxide phase adjacent to the substrate and a subsequent growth of more equiaxed grains of another oxide phase on top of the first oxide layer (Fig. 2a). At 450 °C columnar grains are observed in magnetite, whereas wüstite is the thick, columnar layer at 650 °C. The magnetite layer growing at 450 °C is, however, duplex with a globular seam arising at the interface with the substrate. This thin globular layer appears to stem from a zone of magnetite nucleation, thus inheriting the out-of-plane orientation of single crystal substrates during the early oxidation (Fig. 2b). Hematite, which grows as randomly oriented platelets on magnetite, was shown to have any significant preferential orientation of growth.

The typical time-evolution of the average growth stresses in iron oxides forming at 450 °C is displayed in Fig. 3a. Owing to the weak macroscopic texture of the iron oxides, the scales growing on iron single and polycrystals behave similarly. In both, hematite as well as magnetite, compressive stresses rapidly develop as a result of oxide growth within the grain boundaries of the fine-grained oxide scales grown at 450 °C. In hematite, the topmost layer, the compres-

sive stresses are higher than in magnetite due to its greater unit cell volume and the increased oxygen mobility inside hematite. With increasing oxidation time, the internal stresses in both subscales start to relieve due to a competitive process between the oxide growth strains and substrate creep.

The internal stress situation in iron oxide scales growing at 650 °C strongly depend on time (Fig. 3b), indicating severe scale damage and therefore unsatisfactory oxidation resistance. Wüstite develops internal tensile stresses of up to 300 MPa after 5h, which are followed by a sudden drop to high compressive stress values of about -500 MPa after 7h, probably due to cracking and subsequent oxide formation within voids. Tensile stresses can arise in the non-stoichiometric wüstite as a result of vacancy and porosity gradients across the scale, which is accompanied by lattice parameter variations. Magnetite shows the opposite internal stress development as wüstite, with initially increasing compressive stresses of up to -750 MPa after 4h of oxidation, which are followed by a sudden stress decrease to the same compressive stress values of wüstite, also caused by inner layer damage. Hematite on top shows a different trend, starting with high internal compressive stresses of -1.0 GPa,



**Fig.3:** Time evolution of the average growth stresses during oxidation of an iron single crystal (110) at 450 °C (a) and at 650 °C (b).

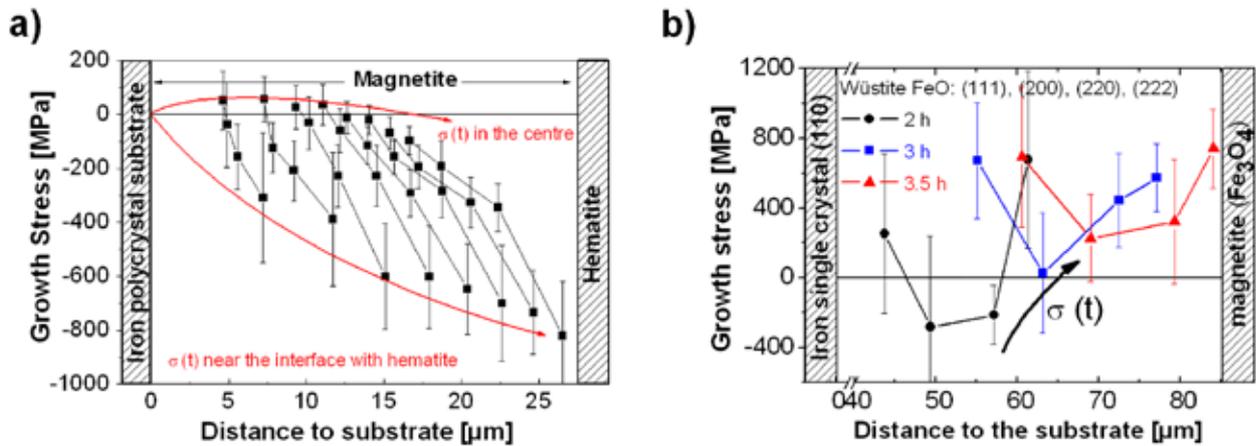


Fig.4: Time evolution of the growth stress gradients in the magnetite layers growing at 450 °C (a) and within wüstite at 650 °C (b).

followed by a permanent and slow stress relieving to compressive stresses similar to those of wüstite and magnetite. This behavior suggests that hematite remains unaffected on top during the oxidation.

The in-depth distributions of growth stresses could be assessed for the thicker sublayers growing at 450 and 650 °C. Magnetite evolves at 450 °C (Fig. 4a) stress gradients which do not significantly change with the oxidation time, reinforcing that layer damage does not take place within the first 10h of oxidation. Increasing compressive stresses are observed towards the interface with hematite. This is caused by the intergrowth of hematite platelets inside the magnetite layer since the early oxidation. Later the increased diffusion of iron along grain boundaries leads to more significant magnetite formation within voids and grain boundaries of the outer regions, maintaining therefore higher compression close to the upper interface. Towards the substrate stress relief is observed since the initial oxidation due to the growth of magnetite via a non-stable diffusion layer of wüstite [25,26] and the creep of the iron substrate. At 650 °C (Fig. 4b) the iron oxide scale is not protective any longer and as a result wüstite undergoes stress gradients which strongly vary with the oxidation time. The results further confirm that within this high-temperature oxidation regime tensile stresses partly arise due to the inhomogeneous distributions of pores as well as of vacancies inside wüstite.

After cooling from the oxidation temperature down to RT, the thermal expansion mismatch between the substrate and the iron oxides determines the residual stress state. Scale damage due to the cooling process from 650 °C causes stress relief to lower compressive stresses in wuestite and to small tensile stresses in magnetite. These low tensile stresses in magnetite result from a significant increase of thermal expansion occurring above 500 °C which is caused by a change from the inverse spinel to the normal spinel structure. After oxidation at 450 °C stress

relief takes place to a greater extent only within the topmost hematite layer.

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# Microstructure and Mechanical Properties of Overlap Friction Stir Welds between AA6181-T4 Aluminium Alloy and HC340LA High Strength Steel

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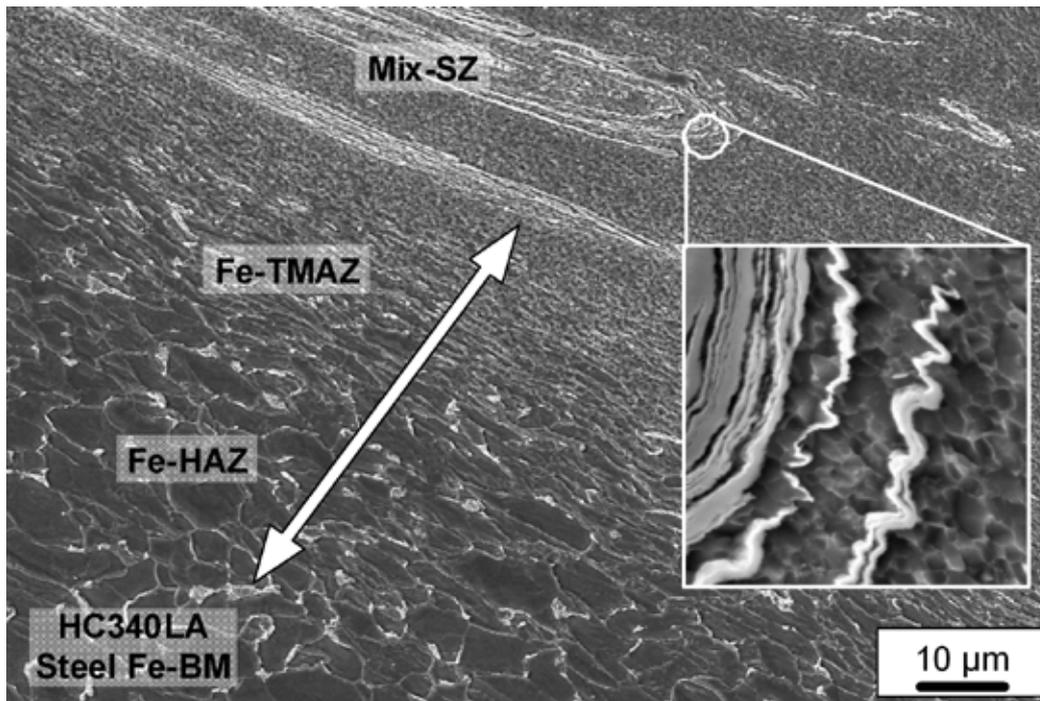
The need for energy saving and pollution reduction has a strong impact on material selection for transportation systems. In the automotive industry in particular there is an increasing use of light-weight materials such as high strength steels, Al alloys and Mg alloys. The introduction of Al alloy parts into a steel car body necessitates the development of reliable and cost efficient joining methods. Substance to substance joining of steels to Al alloys has the benefit of high versatility and cost effectiveness. However, it still presents a challenge, both from the point of view of scientific interest and industrial application. This is due to the strong differences in the melting intervals, the large differences in the physical (e.g. thermal conductivity, thermal expansion) properties and the mechanical properties of steels and Al alloys, and in particular due to the extremely low solubility of Fe in Al which promotes the formation of Al-rich  $Fe_xAl_y$  intermetallic phases. These Al-rich intermetallic phases, namely  $FeAl_2$ ,  $FeAl_3$  and  $Fe_2Al_5$  are known to be brittle and thus are detrimental to the mechanical properties of the joints.

Solid state welding processes naturally avoid solidification and, thus, have particular advantages for joining dissimilar materials with very different melting intervals or materials prone to hot cracking [1,2]. Among pressure welding methods, friction welding has gained increasing application within the last years which is mostly a result of the development of the friction stir welding (FSW) technique [3-4]. The feasibility of FSW of steels was demonstrated as early as 1999 [5], but appeared not to be cost effective at that time, due to strong tool wear. It has been shown that friction stir welds of mild steels [6] or stainless steels [7,-9] to Al alloys can be produced and that the comparatively low heat input in friction welding and friction stir welding reduces or even suppresses the formation of brittle intermetallic phases [10-12]. However, there are only a very limited number of studies concerned with friction stir welds of steels to Al alloys and these mainly focus on process parameters, tool materials or on intermetallic phase formation, lacking a systematic investigation of joint microstructure and mechanical properties.

Here, overlap friction stir welds between AA6181-T4 and the high strength steel HC340LA are investigated. This study focuses on the characterization of the microstructure formation resulting from the friction stir welding process. The characterization is performed using scanning electron microscopy (SEM), electron backscattered diffraction (EBSD) and transmission electron microscopy (TEM). Major emphasis is further placed on the assessment of the  $Fe_xAl_y$  intermetallic compound formation in the mixed stir zone and on understanding the relation between joint microstructure and its mechanical properties. Based on fractography, a possible failure scenario for the AA6181-T4 and the HC340LA steel friction stir welds is proposed.

**Experimental Details.** AA6181-T4 and HC340LA steel sheets (size:  $150 \times 220 \times 1.5 \text{ mm}^3$ ) were friction stir welded in overlap configuration at GKSS Forschungszentrum, Geesthacht, Germany, using a Tricept robot equipped with a mechanical clamping table. The tool material was a UHB Marax ESR (1.6358) alloy. A tool consisting of a 13 mm diameter concave shoulder and a 5 mm cylindrical threaded pin was selected for this study. Microstructure and local texture of the dissimilar friction stir welds were investigated using scanning electron microscopy (SEM) in combination with electron back scattered diffraction (EBSD). Transmission electron microscopy (TEM) techniques were used to investigate the morphology and phase composition of the interface region. Microhardness tests and shear tests were performed in order to assess the mechanical properties of the joints. Microhardness was measured on the specimen cross-section according to DIN 50359-1:1997, Universal Hardness (HU) standard, applying a 0.02 N load. Microhardness measurements were performed on the AA6181-T4 base material, the HC340LA steel base material and the different zones of the weld. Specimens for shear tests with a gauge section of  $3 \times 10 \times 45 \text{ mm}^3$  perpendicular to the welding direction were extracted by spark erosion. After the shear tests, fracture surface analysis for determining the failure mechanisms was carried out in the SEM.

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**Fig. 1:** Secondary electron micrograph of the cross-section of the friction stir processes zone. An arrow indicates a gradient of the grain size from the base material (BM), heat affected zone (HAZ), thermomechanically affected zone (TMAZ) and the mix-stir zone (Mix-SZ) resulting from accumulated strain.

**Results and discussion.** A macroscopic examination of the top side of the overlap weld reveals a good surface quality of the stirred zone and neighbouring areas within the AA6181-T4. Both the surface of the weld and its cross-section do not show any macroscopic defects.

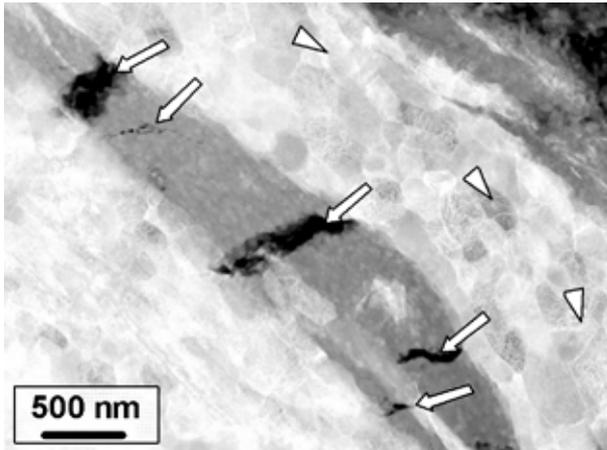
Performed EBSD investigations revealed presence of the expected different weld zones: in the AA6181-T4 (Al-BM): heat affected zone (Al-HAZ), the thermomechanically affected zone (Al-TMAZ), the stir zone (Al-SZ), the mixed stir zone (Mix-SZ) and in the HC340LA steel: the thermomechanically affected zone (Fe-TMAZ), the heat affected zone in the steel (Fe-HAZ) and the steel base material (Fe-BM). Obtained maps of crystallographic orientation distributions (texture) show the inhomogeneous texture distribution within the friction stir weld (slight difference in texture between the advancing and retreating sides of the joint in the AA6181-T4). In the HC340LA steel significant differences between the texture in the base material and the Fe-HAZ were not observed. The EBSD analysis of the grain size distribution demonstrates that in both joined components a heat affected zones are not clearly visible; the heat input of the applied FSW process appears to be not high enough to produce grain growth.

Due to the strong plastic deformation in the vicinity of the pin in the Fe-TMAZ the grains and various stages of subgrain development can be observed in Fig. 1. Although subgrain formation occurs, significant

differences between the texture in the base material and the Fe-HAZ are not observed. The gradient in grain size between the Fe-BM and the steel in the mixed stir zone is much stronger than the gradient in grain size between the Al-BM and the Al-SZ.

Steel inclusions were found incorporated into the Al-SZ region in particular at the retreating side of the pin. Compared to the Al-SZ on the advancing side, the amount of steel particles in the Al-SZ on the retreating side is higher and they are larger (up to 50  $\mu\text{m}$  diameter). The difference in the incorporation of steel particles can be related to material flow in the FSW process. Material flow is driven by both the rotation and the travelling of the tool along the weld. On the advancing side, the direction of the tool rotation coincides with the travelling direction, while on the retreating side the rotation of the tool is contrary to the travelling direction. Material flow therefore appears to be smooth on the advancing side. On the retreating side the opposite movement directions result in steel material removal by the tool and in the transport of steel particles into the Al-SZ where they remain embedded in the Al matrix (Fig. 3). The microstructure of the steel inclusions in the Al-SZ is the same as the microstructure of the steel within the Mix-SZ. The steel inclusions are surrounded by fine bands of intermetallic compounds.

The Mix-SZ develops in the steel adjacent to the Al-SZ, it extends about 40  $\mu\text{m}$  into the HC340LA steel. The main part of the Mix-SZ is characterized by fine equiaxed  $\alpha\text{-Fe}$  (ferrite) grains with an average size



**Fig. 2:** Results of TEM investigations performed in high angle annular dark field (HAADF) imaging mode (due to differences in density the HC340LA steel appears bright, the intermetallic compounds appear grey and the AA6181-T4 appears dark in the HAADF images) reveal presence of micro-cracking in the  $Fe_2Al_5$  intermetallic compound. The cracks are oriented perpendicularly to the undergoing plastic deformation generated by the rotating pin (arrows pointing right down).

of about  $0.27\ \mu\text{m}$  and intermetallic  $Fe_xAl_y$ -compounds formed during the FSW process. The intermetallic compounds are present along the entire interface between the HC340LA steel and the AA6181-T4 alloy. They form thin stripes of about  $0.2\ \mu\text{m}$  to  $1\ \mu\text{m}$  thickness, which intrude up to  $40\ \mu\text{m}$  into the steel (Fig. 1). In friction stir welding, it is generally agreed that thermal energy is generated by contact friction between the shoulder and the base material and through adiabatic heating (i.e. shear processes) around the pin. In the overlap joints examined here, the pin intrudes only very slightly into the HC340LA high strength steel. Softened AA6181-T4 is transported towards and pressed against the exposed HC340LA steel region rubbed by the pin. High local temperatures and high axial pressures promote an accelerated diffusion process leading to metallic bonding between the joint partners. A part of the softened AA6181-T4 is pushed upwards (counter-flow), due to the continuous downward flow imposed by the rotating threaded pin. As a result, some of the detached steel particles are moved away from the bonding line. The chemical reaction and diffusion between the Al alloy transported into the steel and the steel result in the formation of the thin stripes with intermetallic compounds.

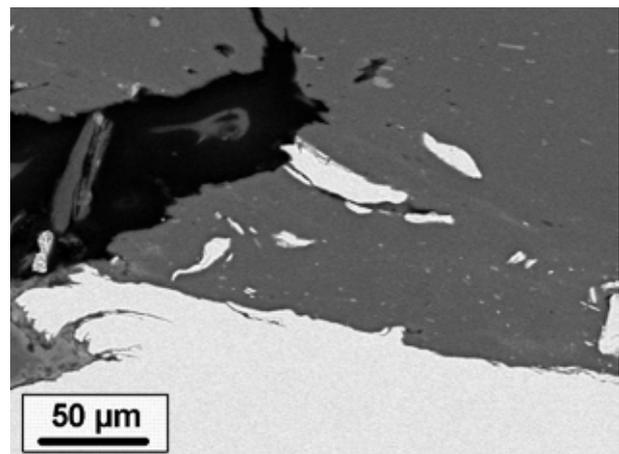
In terms of the binary Al-Fe equilibrium diagram in the Al-rich corner, the three phases  $FeAl_3$  ( $\theta$ -phase),  $Fe_2Al_5$  ( $\eta$ -phase) and  $FeAl_2$  ( $\xi$ -phase) might be expected to be present in the intermetallic phase layer. All these phases in the Al-rich corner do not differ strongly in their thermal stability and their chemical composition. Thus, in joints where layers of intermetallic phases between steel and

aluminium are as thin as those encountered here, only TEM diffraction patterns allow a reliable phase identification. Here, the diffraction patterns taken revealed the intermetallic compounds as  $Fe_2Al_5$ . TEM bright field images showed the grain size of the intermetallic compound to be smaller than  $50\ \text{nm}$ .

Additional TEM investigations were performed in high angle annular dark field (HAADF) imaging mode (due to differences in density the HC340LA steel appears bright, the intermetallic compounds appear grey and the AA6181-T4 appears dark in the HAADF images). In the  $Fe_2Al_5$  intermetallic compound, some nano-sized cracks (marked by arrows in Fig. 2) were observed, which are oriented perpendicularly to the undergoing plastic deformation generated by the rotating pin.

The microhardness measurements were performed horizontally and in perpendicular direction to the cross-section of the joints. While the microhardness of AA6181-T4 is not significantly different in the Al-BM, Al-HAZ, Al-TMAZ and the Al-SZ, the microhardness profile of the HC340LA high-strength steel measured along the Fe-BM, Fe-HAZ and Fe-TMAZ close to the Mix-SZ, shows a hardness increase from  $2300\pm 50$  HU plastic (for the Fe-BM) to  $3500\pm 50$  HU plastic in the Fe-TMAZ. The hardness increase in the Fe-TMAZ can be attributed to strain hardening due to the deformation introduced by the rotating pin.

The mechanical properties (overlap shear test) of the samples tested in the as-welded condition compared to the mechanical properties of friction stir welded overlap AA6181-T4 joints reveal an efficiency of about 73 % of the dissimilar AA6181-T4 to steel HC340LA friction stir welds. Fracture of the dissimilar



**Fig. 3:** The microscopic analysis of the fractured surface shows presence of a high number of detached steel inclusions in the AA6181-T4 stir zone near the interface to the mixed stir zone (retreating side). Rupture in shear tests always occurs on the retreating side of the AA6181-T4 stir zone where the steel inclusions are present. The amount and the size of these steel inclusions apparently determine the mechanical properties of the entire joint.

friction stir welds always occurred on the retreating side of the Al-SZ. The force displacement curves obtained on different friction stir welds produced with identical process parameters are very similar, indicating a good reproducibility of the process.

Macroscopic examinations of the surfaces of samples fractured in the shear tests were performed in order to assess the origin of specimen failure in the shear tests and in order to identify which fracture mechanism occurred (Fig. 3). SEM analysis reveals that failure started in the Mix-SZ on the retreating side and propagated into the Al-SZ. Fig. 3 shows the retreating side microstructure where the steel parts are sheared off the Fe-BM. These fine-grained steel inclusions are surrounded by the intermetallic compound. Upon mechanical loading and plastic deformation, the steel inclusions act as stress risers and are thus very likely to initiate crack formation. Therefore, the amount of steel particles incorporated into the Al-SZ presumably determines the mechanical properties of the entire joint. In comparison to the steel inclusions incorporated at the retreating side of the weld, the formation of intermetallic thin layers and fine grained  $\alpha$ -Fe in the Mix-SZ appears to be less critical to joint failure. The fine-grained intermetallics may even offer an additional bonding mechanism to mechanical interlocking. Crack propagation slows down when the crack reaches regions that are poor or free of intermetallic compounds. Fracture within the fine grained Al-SZ appears ductile. Fracture thus starts of as brittle fracture (in the intermetallic compound where the fracture started) and proceeds as ductile fracture (the fracture propagation in the Al-SZ).

**Conclusions.** Sound overlap friction stir welds between the AA6181-T4 and the HC340LA steel were produced.

- The maximum force of the friction stir welded joints obtained in shear test amounts to 73 % of the forces obtained for respective welds of the AA6181-T4 base material.
- In the AA6181-T4 stir zone near the interface to the mixed stir zone, detached steel parts of about 50  $\mu$ m size are observed as inclusions in the AA6181-T4.
- Grain refinement and defect formation in the Fe-TMAZ result in significant strain hardening.

- Intermetallic phase layers of about 0.2  $\mu$ m thickness were observed in the AA6181-T4 stir zone and the mixed stir zone. They form thin stripes with nano-sized grains. In the mixed stir zone the intermetallic phase layers alternate with very fine recrystallized steel grains. In the AA6181 stir zone, the steel inclusions are surrounded by thin layers of intermetallic compounds.
- Rupture in shear tests always occurs on the retreating side of the AA6181-T4 stir zone where the steel inclusions are present. The amount and the size of these steel inclusions apparently determine the mechanical properties of the entire joint. In comparison the steel inclusions on the retreating side of the Al-SZ, the intermetallics formed even appear to add to the bonding by mechanical interlocking.

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## In-situ Damage Characterization during Creep by Microtomography

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Department of Material Diagnostics and Steel Technology

Present work reports new experimental results regarding damage evolution during high temperature creep of a brass alloy. The results have been obtained with the nondestructive microtomography technique developed at synchrotron sources, which made possible for the first time to monitor in-situ the development of single cavities with sizes in the micrometer range. Experimentally determined cavity growth rates are in good agreement with existing theories.

Motivation of our work is related to understanding of damage development in metals subjected to mechanical loads at high temperatures. This knowledge helps predicting creep lifetime of many engineering devices and components having an important economical implication. There are several theories predicting cavity nucleation and growth as a function of applied experimental conditions, their main achievements being summarized in review articles [1,2] and in the book of Riedel [3]. Experimental characterization of damage is usually restricted to two-dimensional (2D) metallographic sections using light- or scanning electron microscopy. Due to their destructive nature and 2D character these methods are inadequate for a damage characterization allowing a direct comparison with theories. Furthermore, due to the detection limit of the techniques, average cavity measures (like average cavity diameter) are not representative for the ensemble of cavities and cannot be used as basis of comparison. To obtain adequate experimental data we have used the method of X-ray microtomography [4-6], which has many advantages over the metallographic techniques. Firstly, it is free of sample preparation artefacts and delivers a 3D characterization of cavities' shape and spatial distribution. Secondly, based on its nondestructive nature the constraint related to the resolution limit of the method can be relaxed. In-situ tomographic reconstructions combined with image correlation techniques make possible monitoring the volumetric evolution of single cavities as a function of time. It is known, however, that cavity growth rates might depend on the actual cavity volume [1,7] so that the interpretation of tomographic results could be also affected by the resolution limit of the method. This has the consequence that the comparison with theoretical models can be performed only for the detected size

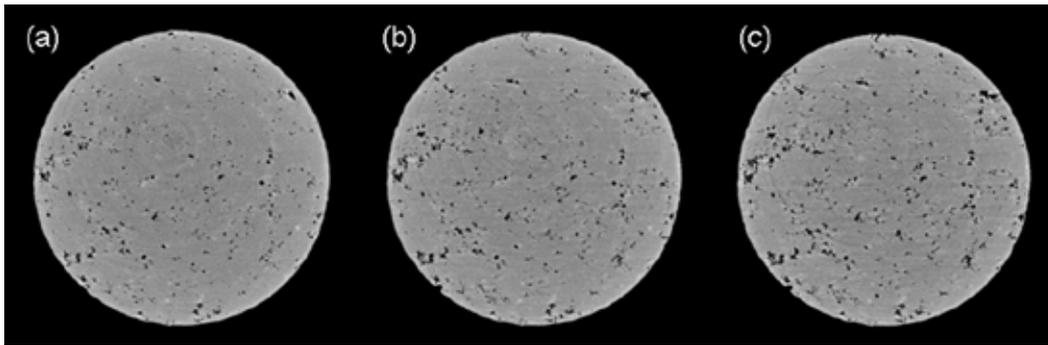
range. In spite of this limitation the microtomographic results are still of high significance because they allow for the first time a direct comparison between experiment and theory.

Experimentally the CuZn40Pb2 brass alloy was investigated, which contains three phases,  $\alpha$ -brass,  $\beta$ -brass and lead-rich particles situated at grain boundaries [4]. The tensile creep test was performed with a miniaturized creep machine (Fig. 1) placed on the goniometer rotating the sample for the tomographic scan. The load was supplied by two springs and measured with a load-cell. An inductive displacement transducer was also used to monitor the displacement of the top of the sample during creep. The high temperature (375°C in present case) necessary for creep was obtained with two heating coils placed at the bottom and the top of the sample. Using two coils assures a homogeneous temperature distribution in the middle part of the sample irradiated by X-rays. In previous investigations the arrangement with only one coil has been used also, with the



Fig. 1: Miniaturized creep testing device for in-situ tomographic measurements.

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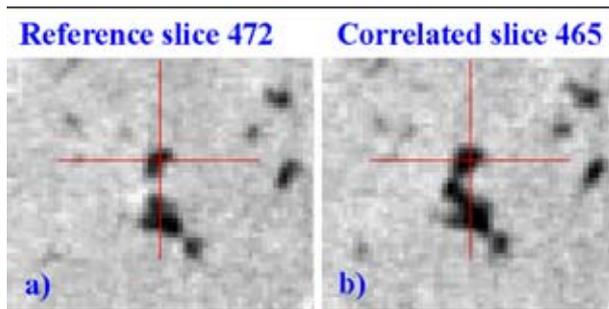
**Fig. 2:** Example of slice registration in two subsequent reconstructed volumes using phase correlation: (a)  $t = 389$  min, (b)  $t = 416$  min, (c)  $t = 440$  min.

aim to impose a constant temperature gradient in the reconstructed volume. These experiments have shown that the tomographic method makes possible the determination of the activation energy of steady-state creep on a single specimen [8]. Results obtained on one small sample with a temperature gradient are in good agreement with literature data obtained on many macroscopic samples deformed at different temperatures [9].

In-situ tomographic measurements during creep were performed at beamline ID15A of the European Synchrotron Radiation Facility (ESRF) using a high energy beam (~80 keV) with a large bandwidth (~50%). The absorption radiographs were recorded with a fast charge-coupled device (CCD) of 1.6  $\mu\text{m}$  effective pixel size. The time necessary for obtaining a complete tomogram was about 3 min, which allowed reconstructions of good quality; material displacements in the illuminated volume during this time remaining below the resolution of the equipment. The typical creep lifetime of the investigated specimens was around 10 hours.

To achieve our aim - following the volume evolution of single pores - a two-step image correlation algorithm was developed in MATLAB. The algorithm is based on the assumed axially symmetric property of the deforming material during creep, the symmetry axis coinciding with the direction of the external load. According to this, the first image correlation step was applied to entire material slices lying perpendicular to

the stress and it allowed quantifying the new position of a selected slice in successively reconstructed volumes. The correlation between 2D images was calculated with the phase-correlation method, which usually allows image registration with subpixel accuracy [10]. Figs. 2a, 2b and 2c show such a slice tracking. Visual inspection of registered slices indicates that cavities (dark features in the image) present in slice (a) appear in slice (b), too. Similarly the voids of slice (b) can be recognized in slice (c) as well. The correlation information can be now used to predict the new center of mass (CM) position of existing voids. Figs. 3a and 3b show enlarged images of voids matched obtained in this way. The red crosses in Figs. 3a and 3b indicate the CM of the cavity in the reference slice and its predicted position in the correlated slice, respectively. It is visible that the predicted position of the CM falls inside the selected void, enabling to monitor its development. This method allowed tracking of about 80% of the cavities and indicates that the initial assumption of homogeneous material flow can be used as a zero order approximation. Smaller pores situated near the border of the slice were lost due to their larger lateral displacements. To cope with this problem a second correlation step was implemented using a two-dimensional region of interest (ROI) centered on each selected pore. The two-step algorithm is slower but provides very accurate results, the proportion of cavities lost during the tracking procedure being less than 0.5%.



**Fig. 3:** Example of cavity detection using the slice correlation algorithm.

Theories of cavity growth predict generally the volumetric growth-rate of single isolated pores [7,11], which means that the comparison between experiment and theory requires detection of cavity coalescence. In order to achieve this, two different methods were developed. The simplest algorithm is based on the ancestor analysis. According to this, a coalescence event is detected when volumes of two cavities from the reference reconstruction have nonzero intersection with the volume of the same single cavity from the correlated reconstruction. Unfortunately this analysis is unable to detect the coalescence of an already recognized cavity with



cavities reaching the detection limit in the time interval between reconstructions. To improve the confidence of coalescence detection a new indicator based on the morphology of the cavity was developed, which can partly account for coalescence events with “invisible” pores. This second parameter, named as “complexity factor”, describes the difference between the actual pore shape and its equivalent ellipsoid [5]. Based on a visually determined criterion, cavities with complexity factor larger than 0.75 were considered as coalesced. From the point of view of growth-rate evaluations cavity coalescence was considered completed when either the ancestor or the complexity criterion was fulfilled at first.

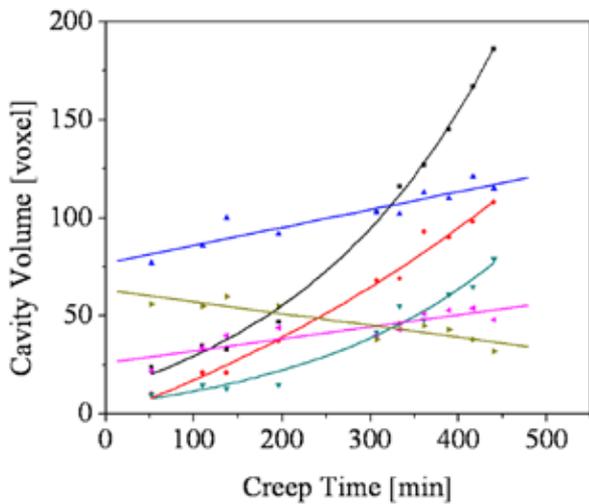


Fig. 4: Volume growth-curves as a function of time for different cavities.

Fig. 4. shows the volume change of several cavities as a function of time. The measurement points can be approximated by a large variety of analytic functions ranging from linear till exponential growth. The proportion of the latter one is, however, very small. Interestingly, voids with decreasing volume were also detected. Since void growth theories restrict their predictions to steady-state creep, only the first four points of the volume growth cures were taken into account and they were fitted by straight lines. The histogram of growth-rates (Fig. 5) obtained from the analysis of about 6200 voids has a mean value of about 0.26 voxel/min (~1 μm<sup>3</sup>/min), which can be compared with predictions of theoretical models. Considering diffusion as main mechanism of void growth, the growth rate can be evaluated based on Dyson’s work [12], which predicts a value of about 2·10<sup>-5</sup> voxel/min. This is much lower than the experimental one indicating the inadequacy of the assumption. Another possibility is to consider that “power-law creep” determines void growth [7]. This mechanism based on plasticity predicts growth-rates, which are dependent on the actual pore volume. The higher the pore volume the higher is its growth rate, a prediction different from that of diffusion theory

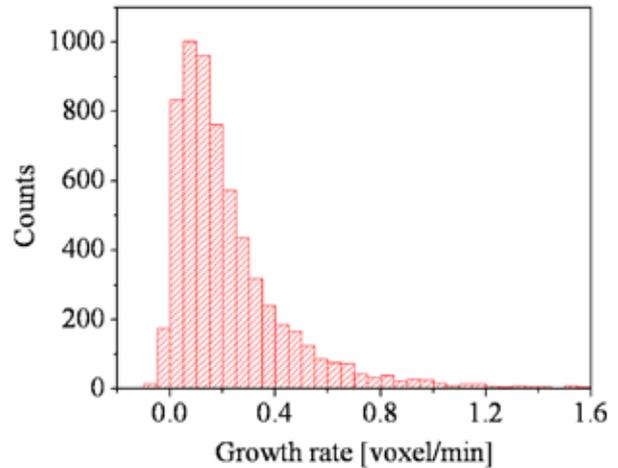


Fig. 5: Histogram of cavity volume growth-rates in steady-state creep.

foreseeing a constant growth rate with time. Based on the measured steady-state strain-rate of the investigated sample ( $\dot{\epsilon}_{ss} = 3 \cdot 10^{-6} \text{ s}^{-1}$ ) and the stress exponent of  $n = 3.4$  taken from the literature [9], the volume growth of detected pores can be calculated according to eq. (1) [7]:

$$V = V_0 \exp \left[ \frac{n+1}{n} \ln \left( \frac{1 - \dot{\epsilon}_{ss} n t_0}{1 - \dot{\epsilon}_{ss} n t} \right) - \dot{\epsilon}_{ss} (t - t_0) \right]$$

where  $V_0$  represents the volume of the pore at time  $t_0$ . This model predicts volumetric growth-rates between  $4.7 \cdot 10^{-3}$  and  $0.19$  voxel/min, which are in good agreement with experimental data, explaining 56% of the histogram shown in Fig. 5. The goodness of eq. (1) in predicting experimental data is shown in Fig. 6. for the case of a selected pore. The theoretical curve, which uses only the pore volume (at stage 2) as input microstructural parameter predicts accurately the volume growth until stage 8, after which the pore grows at a much higher rate. Stages 9

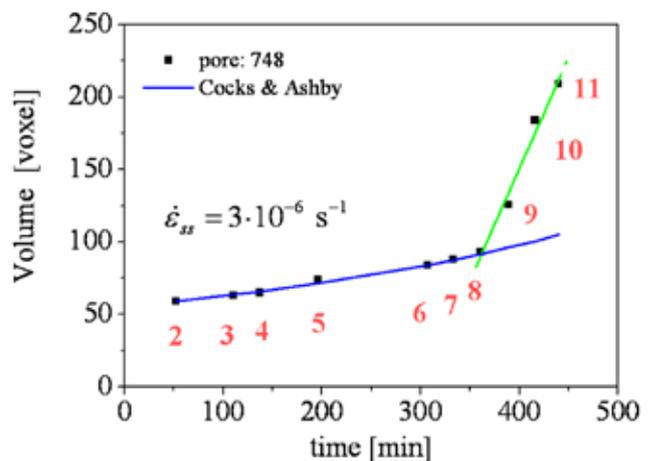


Fig. 6: Volume growth of a selected cavity fulfilling the Cocks and Ashby prediction [7].

to 11 are, however, characterizing accelerated creep, when the assumptions of the theory do not hold.

As creep proceeds, plastic deformation leads to the development of a triaxial stress-state around pores. Based on finite element calculations [13] local triaxiality values of about 1 are usually found near pores. This accelerates their growth and according to refs. [1,7] a multiplying factor of about 2 should be taken into account. The predicted upper limit of the growth rates becomes now about 0.4 voxel/min making the agreement between theory and experiment better. The negative and high growth rates (above 0.4 voxel/min) cannot be explained by the plasticity mechanism. A possible reasoning can be based, however, on the special structure of the investigated material. Since the deformation temperature is higher than the melting temperature of lead, the initial lead-rich particles at grain boundaries become liquid and are wetting the grain boundaries. The existence of the liquid film between grains may favor grain boundary sliding producing higher growth rates of the voids. Experimentally found negative growth rates can be also explained by the wetting phenomena. The thermal expansion coefficient of lead ( $29.3 \cdot 10^{-6} \text{ K}^{-1}$ ) is higher than that of brass ( $20.9 \cdot 10^{-6} \text{ K}^{-1}$ ) which combined with the volume extension of lead during melting leads to high hydrostatic stresses in the lead phase. These stresses promote grain boundary wetting and voids situated near the molten lead can be filled in progressively.

It was shown that volumetric growth-rates of pores during steady-state creep of the CuZn40Pb2 brass alloy are in good agreement with predictions of the Cocks and Ashby model [1,7]. The results are representative for relatively high strain-rates of the

order of  $10^{-6} \text{ s}^{-1}$ , which were allowed by the finite measurement time available at the synchrotron source.

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## Hierarchical Nature and Properties of Biological Chitin-based Nanocomposites: Lobster Cuticle

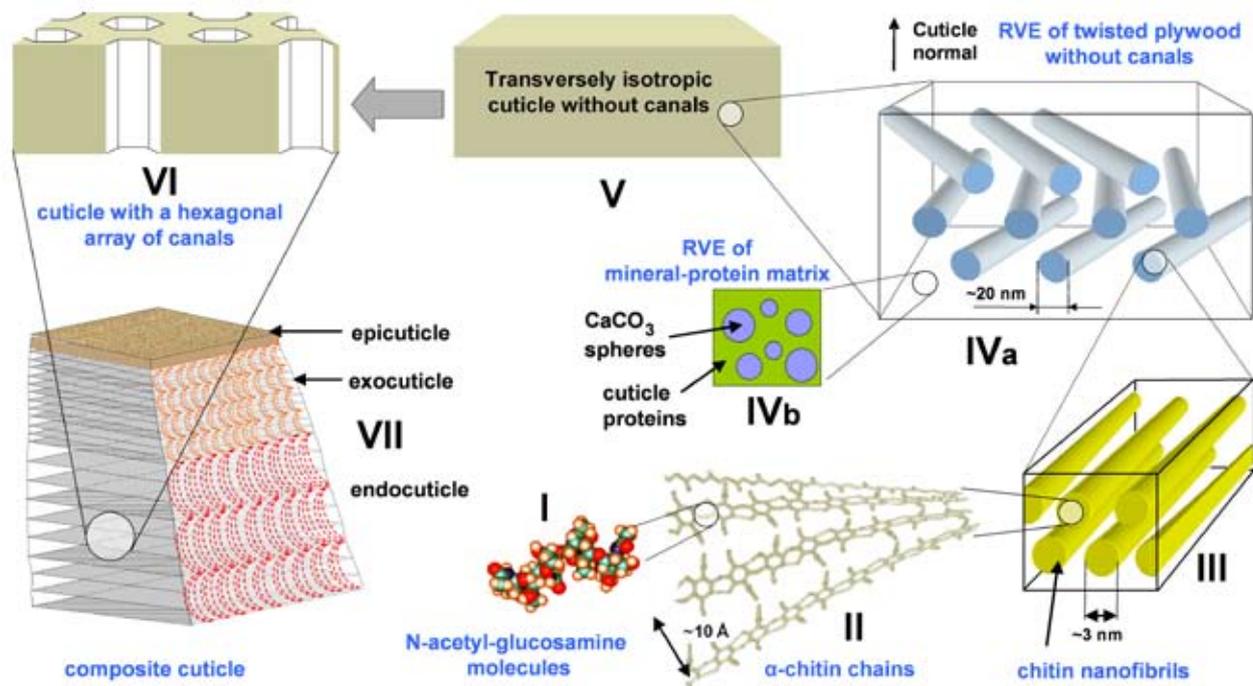
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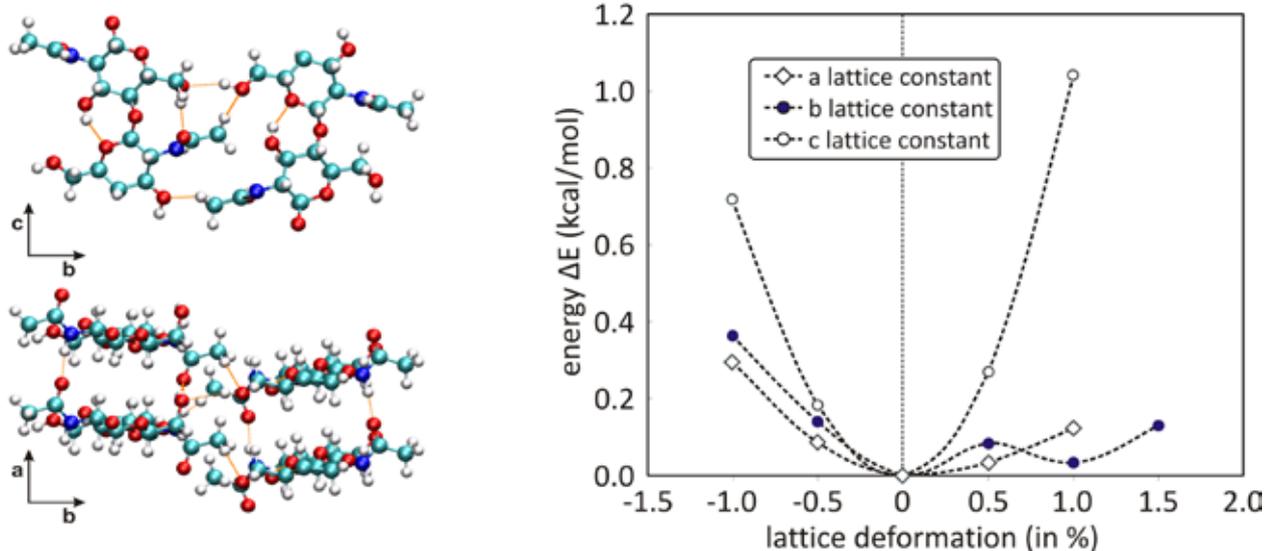
Understanding the structure/property relations in mineralized biological tissues such as bone, mollusc shells, and arthropod exoskeletons at different length scales brings an insight about how nature designs multifunctional structural materials as well as about the evolution of species. Moreover, mineralized biomaterials inspire the elaboration of a next generation of advanced nanocomposites for numerous applications in nanotechnology, structural and tissue engineering, and armour design. In this study we briefly highlight our recently developed multiscale model for the structure/elastic property relations in crustacean exoskeletons. The cuticle of the lobster *Homarus americanus* is taken as a model material because extensive experimental data about its microstructure, texture and mechanical properties that have been recently obtained at our institute [1-4]. These studies showed that the lobster cuticle has a complex hierarchical organization spanning from nano- to macroscale. It consists of the structural

biopolymer chitin in the form of reinforcing crystalline nanofibrils, various proteins, mineral nanoparticles (mostly amorphous calcium carbonate (ACC) but also small amounts of calcite), and water.

A schematic representation of the hierarchical organization and the constitutive modelling of lobster cuticle as well as the generic design principles of crustacean exoskeletons are shown in Fig. 1. These principles are: i) chitin-protein fibers forming a twisted plywood (Bouligand [5]) structure similar to a chiral nematic liquid crystal (Fig. 1 IVa); ii) mineral-protein matrix that accommodates and stiffens the chitin network; iii) multilayer design at macroscopic level consisting of a thin waxy epicuticle, hard exocuticle and somewhat softer endocuticle. In addition, the lobster cuticle possesses a well developed system of pore canals that facilitate biomineralization and reduce its weight. The canals give it a honeycomb-like appearance.



**Fig. 1:** Structure and modelling of the lobster cuticle: (I), (II)  $\alpha$ -chitin properties via *ab initio* simulations; (III) representative volume element (RVE) for a single chitin-protein fiber; (IVa) RVE for chitin-protein fibers arranged as a twisted plywood and embedded in mineral-protein matrix; (IVb) RVE for the mineral-protein matrix. Level (V): homogenized twisted plywood without canals; (VI) homogenized twisted plywood pierced with hexagonal array of canals; (VII) 3-layer cuticle.

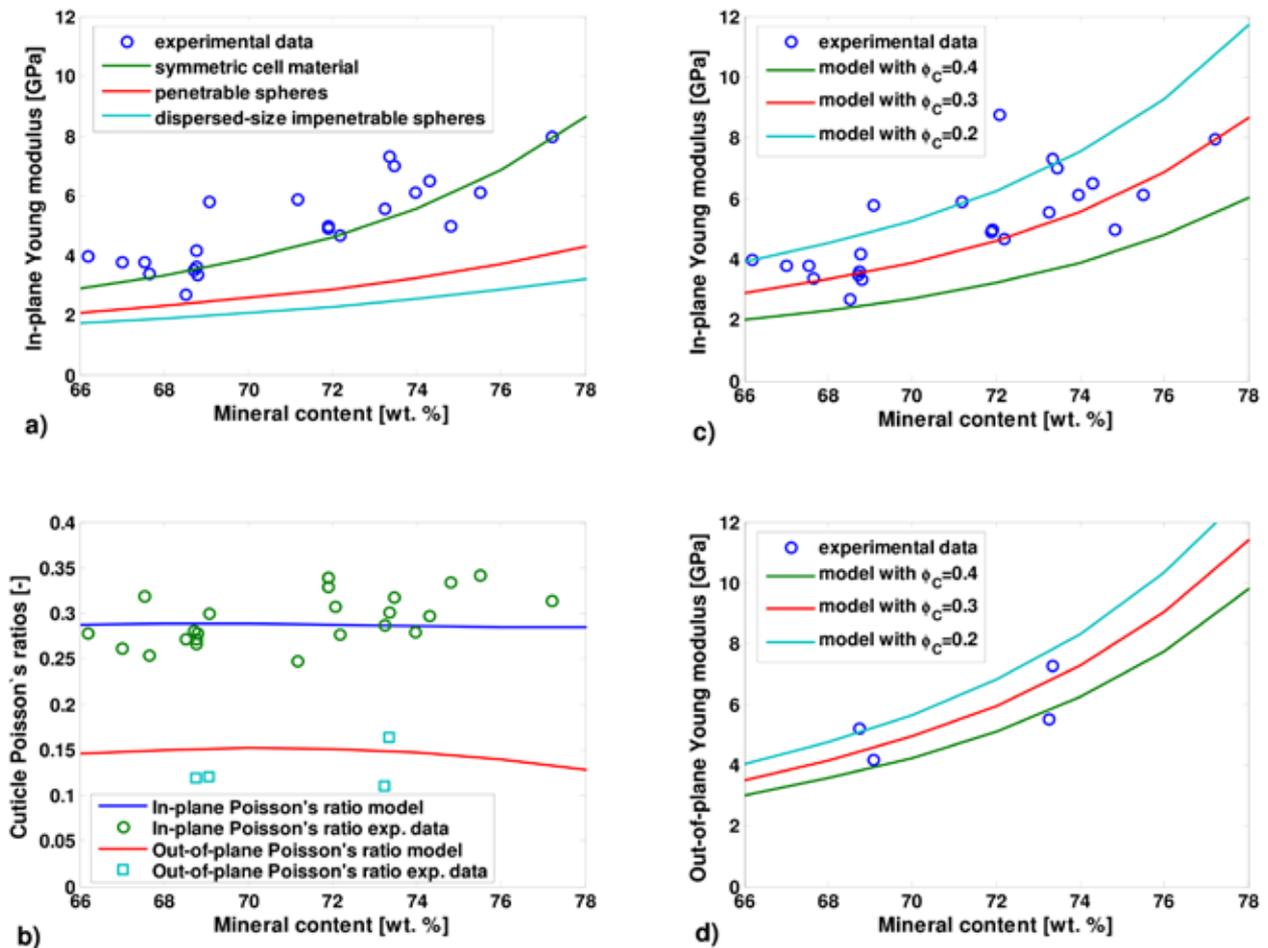


**Fig. 2:** Left: Theoretically determined ground state structure of single-crystalline  $\alpha$ -chitin:  $a$ ,  $b$  and  $c$  (chain direction) denote the orthorhombic unit cell vectors. Different atoms are colored as follows: red – O, blue – N, turquoise – C, white – H; orange lines – H-bonds. Right: total unit cell energy vs. uniaxial strain profiles for deformations along  $a$ ,  $b$ , and  $c$ . Positive (negative) values along the  $x$ -axis denote tensile (compressive) strain. The shallow minimum which appears at  $\sim 1\%$  tensile strain in the lattice deformation profile along  $b$  denotes a metastable configuration and depicts the complex and flexible character of the hydrogen bond pattern.

To obtain the multiscale structure/property relations of the cuticle, we use a hierarchical homogenization method. It includes parameter-free *ab initio* calculations at atomic scale (CM dept.) and bottom-up, step-by-step mean-field homogenization for the higher hierarchy levels (MU dept.). The fundamental level in our multi-scale and multi-disciplinary analysis lies at the atomic scale where the electronic structure and inter-atomic bonding of the studied material determine the elastic properties (Fig. 1 I, II). The identification of the atomic geometry and the structural and elastic properties of crystalline  $\alpha$ -chitin by means of atomistic calculations are challenging: The extended hydrogen bond network, the large system size and conformational variability as well as the high structural flexibility result in a complex Born-Oppenheimer surface with respect to the atomic coordinates. To tackle this problem, a hierarchical computational approach was used that combines valence force field molecular dynamics with tight binding and density functional theory calculations. Based on this approach the atomic structure including the positions of the hydrogen atoms has been identified. Using this structure, the complete set of the lattice and elastic constants of single-crystalline  $\alpha$ -chitin, which is present in all arthropod cuticles, have been determined for the first time. The obtained ground-state structure as well as the response of the bio-crystal to uniaxial strains applied along the orthorhombic unit cell vectors  $a$ ,  $b$ , and  $c$  are shown in Fig. 2. The corresponding elastic constants along the  $c$ ,  $b$ , and  $a$  vectors are  $C_{11} = 119$  GPa,  $C_{22} =$

28 GPa, and  $C_{33} = 24$  GPa, respectively. Based on the elastic tensor, it is evident that  $\alpha$ -chitin is much stiffer in the chain direction  $c$  compared to the transverse directions: This is attributed to the strong covalent bonds which dominate along the chain axis compared to the weak H-bonds which are responsible for the cohesion in the transverse directions.

The effective elastic properties at higher hierarchy levels are found using several homogenization methods [6-8]. These methods require knowledge of the elastic properties of each phase within the composite as well as the volume fractions, shapes, and orientations of each of the dispersed phases. When necessary, Torquato 3-point estimates [8] are used where, in addition to the above parameters, the specific arrangement of the dispersed phases is taken into account via statistical correlation functions. The representative volume element (RVE) of a single chitin-protein fiber (Fig. 1 III) is modeled as a long-fiber composite with isotropic protein matrix and homogenized with a Mori-Tanaka scheme [6]. The Bouligand plywood structure with a system of pore canals is modeled in two steps. First, the effective properties of the bulk plywood composite without canals are found. Then, the pore canal system is taken into account as an array of hexagonal holes piercing the homogenized cuticle. The RVE of the bulk plywood (Fig. 1 IVa) is modeled as an isotropic matrix reinforced with families of parallel chitin-protein fibers perpendicular to the cuticle normal and rotated at a fixed angle with respect to each other. For the



**Fig. 3:** a) In-plane Young's modulus as a function of the mineral-protein matrix microstructure and mineral content; b) Cuticle in-plane and out-of-plane Poisson's ratios for different mineral content; c) In-plane modulus for different canal pores area fractions vs. mineral content; d) Out-of-plane Young's modulus for different canal pores area fractions vs. mineral content. Symbols: experimental data, lines: model predictions;  $\Phi_c$  denotes the in-plane area fraction of pore canals.

effective properties, the dilute approximation is used [7]. The mineral-protein matrix in the bulk cuticle tissue (Fig. 1 IVb) is itself an assembly of amorphous calcium carbonate spheres embedded in cuticle proteins. It is modeled using a Torquato 3-point estimate [8]. The in-plane properties of the cuticle with canal pore system (Fig. 1 VI) are also found with a 3-point approximation for regular honeycombs with thick walls. A simple Voigt average is used to find the cuticle properties along its normal direction.

In Fig. 3 we compare the predicted Young's moduli and Poisson's ratios with data obtained by tension/compression tests on hydrated endocuticle samples. In Fig. 3a it is seen that the most probable microstructure of the mineral-protein matrix is that of a symmetric cell material consisting of impenetrable polydisperse spheres with quasi-random packing. This geometry has additional microstructure correlations that make it very stiff. As a result, the

Young's modulus strongly increases with increasing the mineral content while the Poisson's ratios remain virtually unchanged. An interesting consequence of our analysis is that the biomineralization kinetics in lobster seems to be such that for a given volume fraction of minerals, it produces the stiffest possible cuticle.

An overview of our multiscale simulation/analysis is given in Fig. 4 (see next page). It is seen that locally, the chitin-protein fibers increase the stiffness of the bulk mineralized tissue in the fiber direction by about 50% and the overall cuticle anisotropy decreases from nano- to macroscale. In conclusion, it is found that the dominant factors determining the cuticle stiffness are: i) the mineral content; ii) the specific microstructure of the mineral-protein matrix; iii) the in-plane area fraction of the pore canals, and iv) the chitin-protein fibers reinforcement.

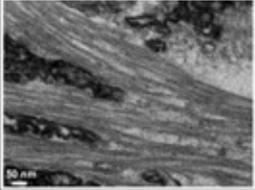
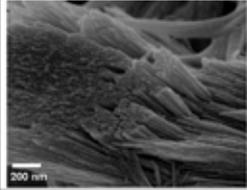
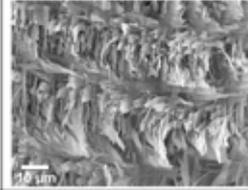
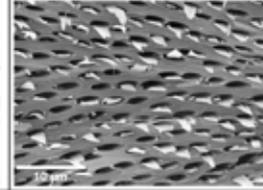
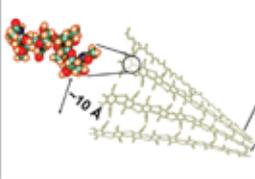
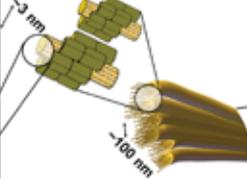
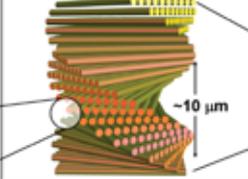
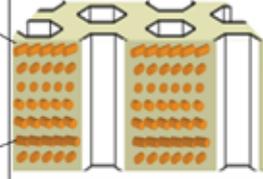
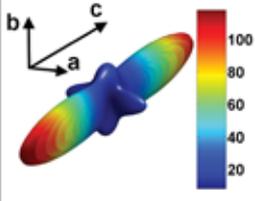
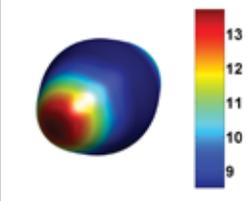
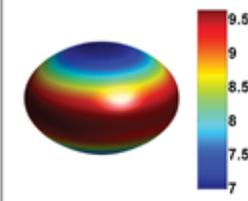
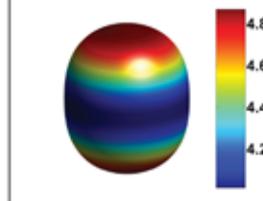
Scale	0.1 nm – 10 nm	10 nm – 100 nm	100 nm – 10 μm	10 μm – 1 mm
Hierarchical structure unit	α-chitin (H-bonded anti-parallel N-acetyl-glucosamine molecular chains)	Mineralized chitin-protein nanofibrils in a planar array	Twisted plywood stack of mineralized chitin-protein planes without pore canals	Twisted plywood stack of mineralized chitin-protein planes with pore canals
Experimental method	Transmission electron microscope	Field emission scanning electron microscope	Field emission scanning electron microscope	Field emission scanning electron microscope
Microstructure				
Schematic				
Simulation method	Ab initio; density functional theory	Mori-Tanaka scheme (chitin-protein fiber); Torquato 3-point scheme (mineral-protein matrix)	Dilute approximation, tensor rotation	Torquato 3-point homogenization
Elastic behavior, 3D map of Young's modulus [GPa] a,b-axis: basal directions of chitin cell c-axis: longitudinal axis of molecule				

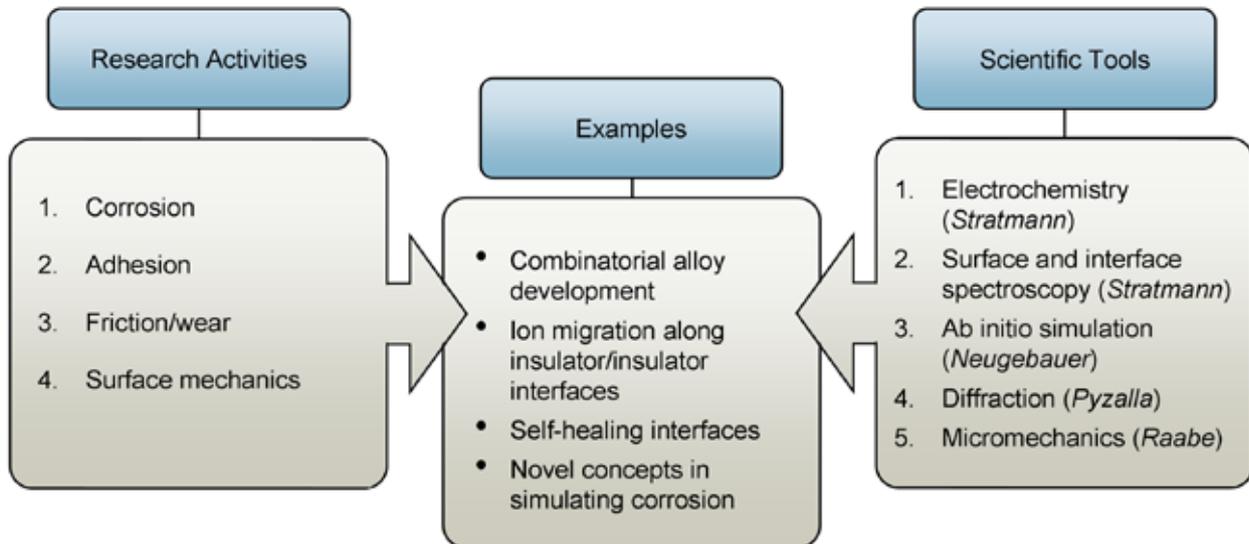
Fig. 4: Overview of key cuticle microstructures at different length scales including the methods used and the predicted anisotropy in the elastic properties for mineral content of 70% wt.

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# Stability of Surfaces and Interfaces



## Selected Highlights 2007/2008

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## High Throughput Characterization in Combinatorial Alloy Development

A.W. Hassel, A.I. Mardare

Department of Interface Chemistry and Surface Engineering

Alloy development must have been purely empiric in ancient times. Later a knowledge based approach guided successfully to the whole range of materials that are presently available. It became more and more difficult to further follow this way, since the effort for making a sufficient number of sample alloys which serve as nodes for the description of materials properties is too high. This is in particular true for multicomponent systems such as ternary or higher alloys in which the possible number of combinations increases with an even higher exponent. New strategies are necessary to cope with this request and high throughput characterisation of composition spread material libraries may be one of them [1].

Samples with a composition spread were produced by thermal evaporation (Al, Fe) or co-sputtering deposition (Hf, Nb, Ta, Ti, Zr) from 2 or 3 sources onto a glass or silicon substrate.

A thorough characterisation of the deposited thin films was performed by FE-SEM and EDX as well as XRD. These scanning methods did not only allow for the determination of the local composition but also for the nanostructure and crystallography of the material.

To allow for an efficient electrochemical characterisation a scanning droplet cell (SDC) was used [2,3]. This technique employs a microelectrochemical cell where only a small electrolyte droplet hanging at the tip of a glass capillary will contact the surface. In this way, small areas can be sequentially addressed,

instead of dipping the entire sample into electrolyte, as usually done when using a classical electrochemical cell. In this way, multiple measurements are possible on the same sample.

Fig. 1 (a) shows a schematic drawing of the SDC used. The body of the cell is comprised of an acrylic block (20mm x 20mm x 10mm) in which four interconnected channels were drilled. Each of the four channels can be sealed using a plastic screw. The present design follows the idea of using a solid state micro-reference electrode ( $\mu$ -RE) built inside of a capillary [4]. The counter electrode (CE) is a high purity (99.999 %) gold band, 1 mm wide, wrapped around the RE capillary as shown in the magnified part of Fig. 1 (a). A 2.5 mm outer diameter capillary is mounted in a screw, representing the electrolyte outlet of the cell. The capillary was previously pulled using a capillary puller and tips with diameters ranging from 1-1000  $\mu$ m (200  $\mu$ m in this specific study) may be obtained using a micro-grinder. For a better definition of the wetted area, a silicone gasket is formed by dipping the tip in liquid silicone followed by drying it in nitrogen stream. The tip of the SDC is pressed against the WE surface so that the silicone will deform slightly, precisely defining the wetted area [5]. If the magnitude of the applied force is small enough, the wetted area can be highly reproducible.

The SDC was mounted on a 3D translation stage actuated by three computer controlled DC-mikes

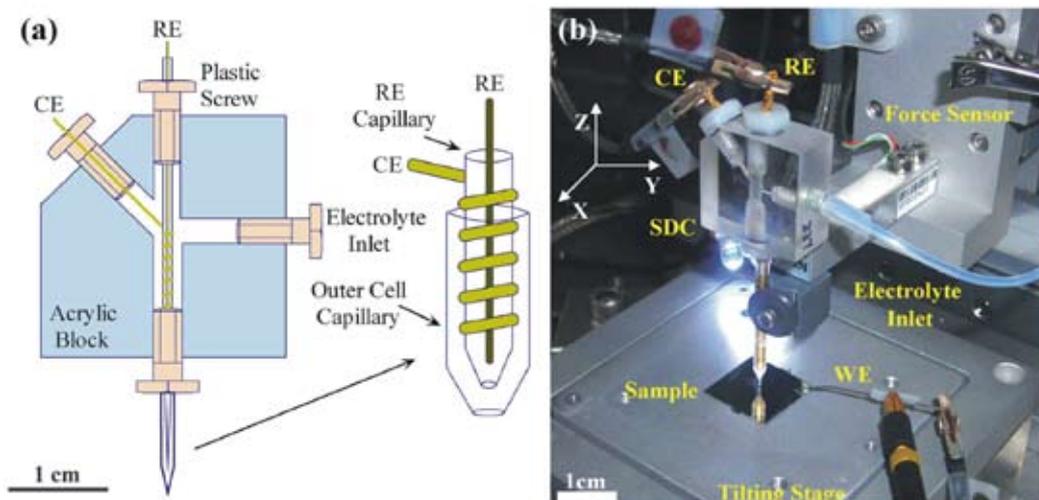


Fig. 1: (a) Schematic drawing of the SDC. (b) Photograph of the automated SDC setup with a 3D scanner.

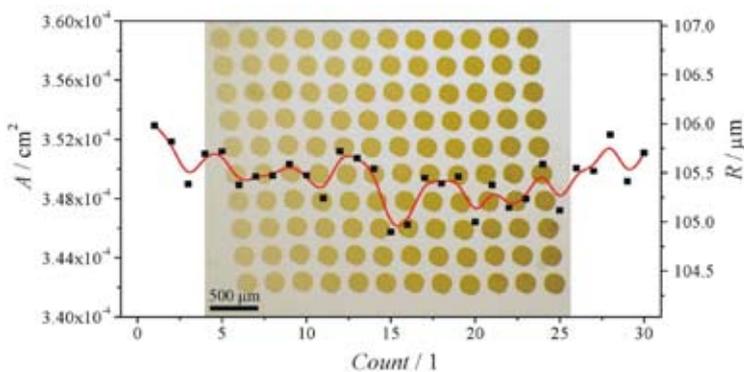


Fig. 2: Matrix of oxide spots anodically grown on a Hf thin film and oxide surface distributions as measured by optical microscopy.

allowing the movement of the cell tip across the sample, in an automated fashion, with a precision of 500 nm [6]. Two video cameras provided live video feed-back from the top, for observing the sample, and laterally from one side, for observing the height of the cell tip above the sample. In Fig. 1 (b) a photograph of the automated SDC mounted to the 3D scanner is shown. The LabView software, self developed for this setup, includes positioning and data acquisition and evaluation modules. These allow the automatic movement of the SDC to predefined positions and computer controlled experimentation in a high throughput manner.

For the SDC used here, a constant force of 3 mN was experimentally found to be sufficient for ensuring an elastic deformation of the silicone gasket formed at the tip of the capillary. A matrix of oxide spots grown at potentials ranging between 4.5 V and 10 V on the surface of a Hf thin film allowed an investigation of the wetting reproducibility. In Fig. 2 these spots can be seen together with the measured areas and calculated radiuses for randomly chosen spots along the growth potential gradient. A deviation of less than 1 μm in the radius defines an error of less than 1 % in the wetted area, demonstrating that the high reproducibility can be achieved.

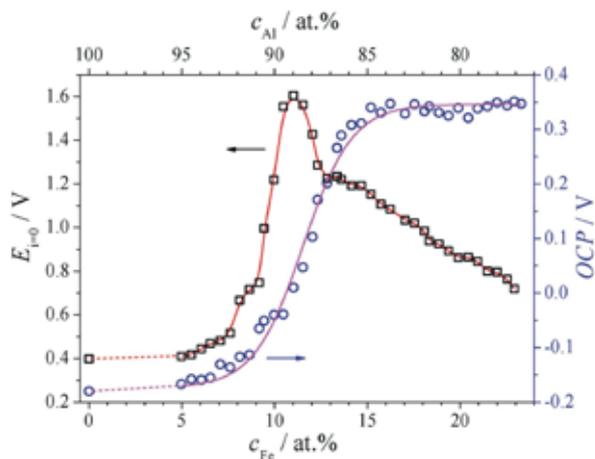


Fig. 3: Open circuit potential and maximum potential for the zero current measured along the compositional gradient on the Al-Fe composition spread.

The automated SDC was used for the study of anodic oxides grown on metallic thin film combinatorial libraries obtained using co-deposition from vapour phase. An Al-Fe compositional spread was studied in the framework light weight steel development. A series of electrochemical measurements were predefined and the combinatorial sample was scanned along the compositional gradient. The measurements included step-wise anodic oxide formation alternating with low and high frequency impedance measurements for oxide's electric resistivity and dielectric constant evaluation. The open circuit potential (OCP) and the potential onset of the anodic oxide formation ( $E_{I=0}$ ) are presented in Fig. 3 as examples of properties mapping in combinatorial libraries. A percolation threshold can be recognized at the concentration corresponding to the inflection point of the OCP curve. In the same time, the zero current potential, which characterizes the thickness of

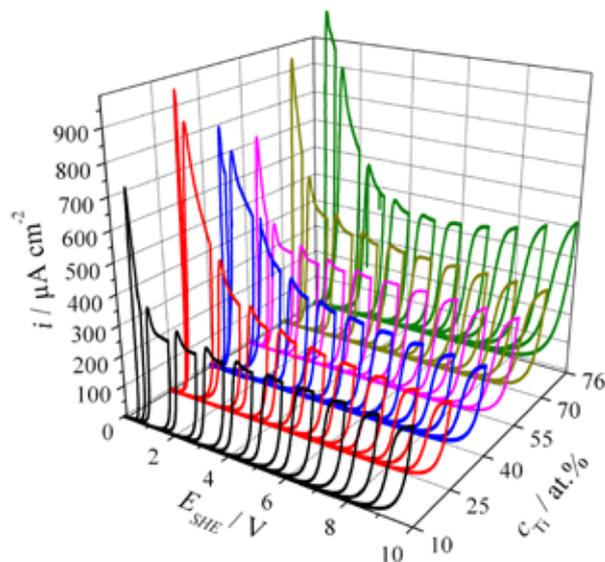


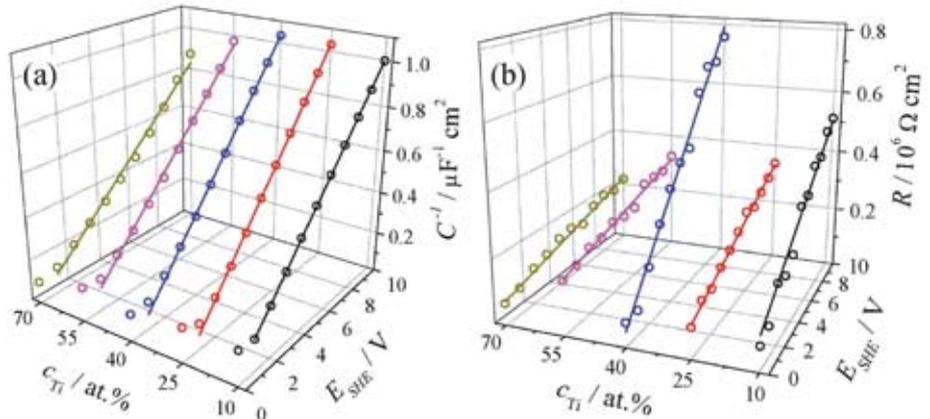
Fig. 4: CVs recorded during the Ta-Ti binary alloys anodizations at different concentrations.

the natural oxide grown on the surface of the parent alloy, shows a peak suggesting much thicker native oxides in the percolation threshold region [1].

Anodic oxides on binary and ternary valve metals alloys (Hf, Nb, Ta, Ti) are another issue. An example of the potentiodynamic anodization sequence measured on the Ta-Ti combinatorial library is shown in Fig. 4. In each of the voltammograms corresponding to the step-wise anodizations from 1 V to 10 V in an anodization series, a current density plateau was observed corresponding to the anodization process dictated by Faraday's charge-mass correlation. The current overshoot indicating a delayed oxide formation can be observed at the beginning of each

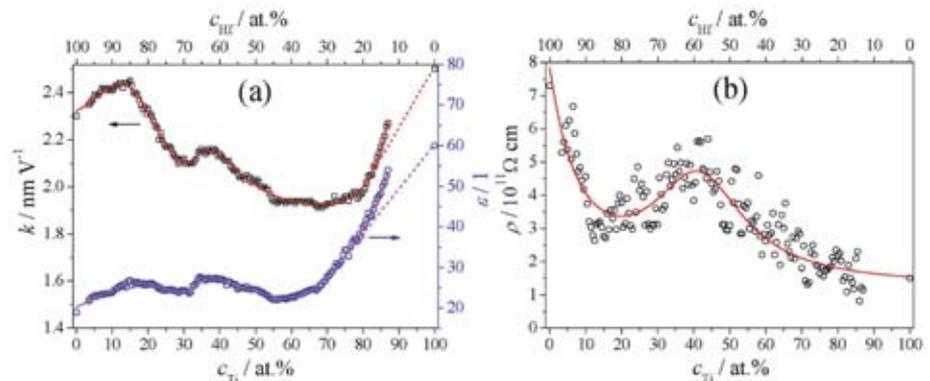


cyclic voltammogram. This is due to charge accumulation in a space charge layer inside the previously grown oxide, as described by the extended high field regime [7]. Due to a constant potential scan rate ( $100 \text{ mV}\cdot\text{s}^{-1}$ ), a weaker field strength increase results for thicker initial oxides, and consequently the current overshoots are less visible at higher potentials.



**Fig. 5:** Inverse capacitance (a) and resistance (b) of mixed anodic oxides grown on Ta-Ti combinatorial library at various formation potentials.

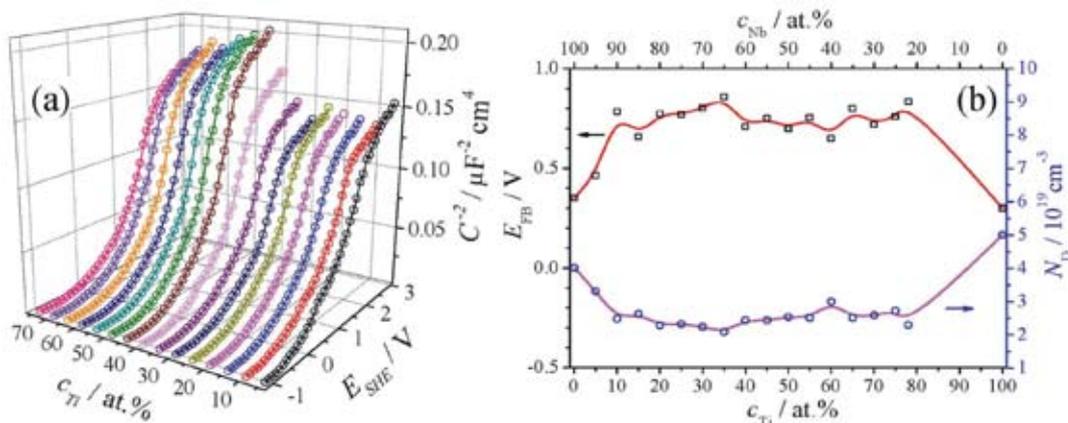
In order to investigate the electric properties of the growing anodic oxides, capacitances and resistances were first analyzed using impedance spectroscopy. As an example, in Fig. 5 the inverse capacitances and resistances of various oxides grown on the Ta-Ti combinatorial library are plotted [8]. Their linear dependence on the oxide formation potential allows directly the evaluation of permittivity and electric resistivity of the oxides.



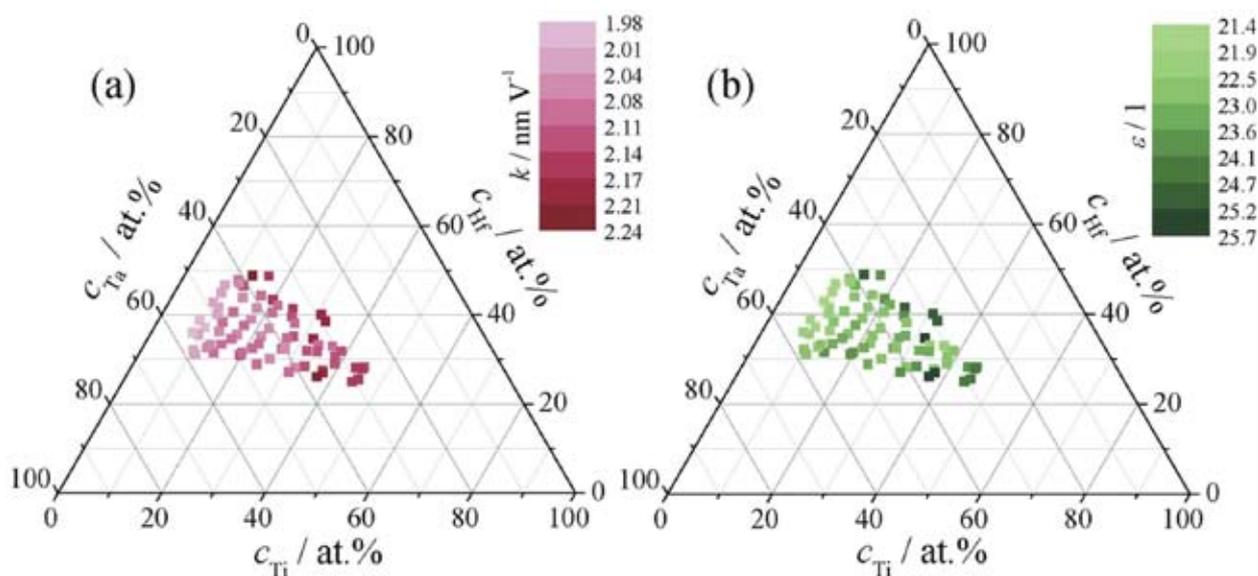
**Fig. 6:** (a) Oxide formation factors and dielectric constants of oxides grown on Hf-Ti alloys. (b) Electrical resistivities of anodic oxides grown on Hf-Ti combinatorial library.

The oxide formation factors together with the dielectric constants and the electric resistivities are shown in Fig. 6 for the oxides anodically grown on the Hf-Ti combinatorial alloys. The mappings show different evolutions of the properties as a function of the parent metal concentrations and can be generally correlated with the properties of the metals revealed by SEM and XRD investigations.

In the cases when the SDC is used for investigating mixed anodic oxides with semiconducting properties, such as the oxides grown on the Nb-Ti compositional spread, Mott-Schottky analysis is applied. Several Mott-Schottky plots corresponding to various metal parent compositions are shown in Fig. 7 (a). The positive slopes of the linear part indicate the presence of type n semiconductors and the linear fits resulted in the mapping of the flat band potential and donor



**Fig. 7:** (a) Mott-Schottky plots for anodic oxides potentiostatically grown at 3V on Nb-Ti library. (b) Flat band potentials and donor densities for anodic oxides grown on the Nb-Ti alloys.



**Fig. 8:** (a) Oxide formation factors of oxides grown on Ti-Ta-Hf alloys. (b) Dielectric constants of anodic oxides grown on Ti-Ta-Hf combinatorial library.

density values along the compositional gradient shown in part (b) of Fig. 7.

When investigating the anodization of ternary thin film combinatorial alloys, the property mapping has to be transformed into ternary plots. An example is shown in Fig. 8 where the oxide formation factor and the dielectric constant of the oxides grown on the Ti-Ta-Hf alloys are presented as colour coded maps for the surface spread of the parent alloys concentrations.

Generally, compositional zones can be defined from the investigation of alloys' microstructure and crystallography. Different zones define different metallic grain structures, such as domains formation (Ta-Ti), grain size variation (Hf-Ti) and shape shifting (Nb-Ti) or formation of amorphous alloys (Al-Fe, Hf-Ta and Ti-Ta-Hf). The permittivities and resistivities of the modified oxides are generally directly influenced by the properties of the alloys' components. Nevertheless, perturbations often appeared creating peaks in the compositional mappings, generally located at the threshold between compositional zones. Surface analytical investigations showed that generally the modified oxides composition deviates from the parent metal composition. An enrichment of Ti in the oxidized state as compared with the amount of Ti present in metallic form in all Ti alloys, was found. Fe and Hf accumulations at the oxide/metal interface were detected for the Al-Fe and Hf-Ta alloys, respectively. These effects were attributed to different ionic transport numbers for the components of the alloys.

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## A New *in-situ* X-Ray Diffraction Setup: Electrodeposition of Zn from Ionic Liquids

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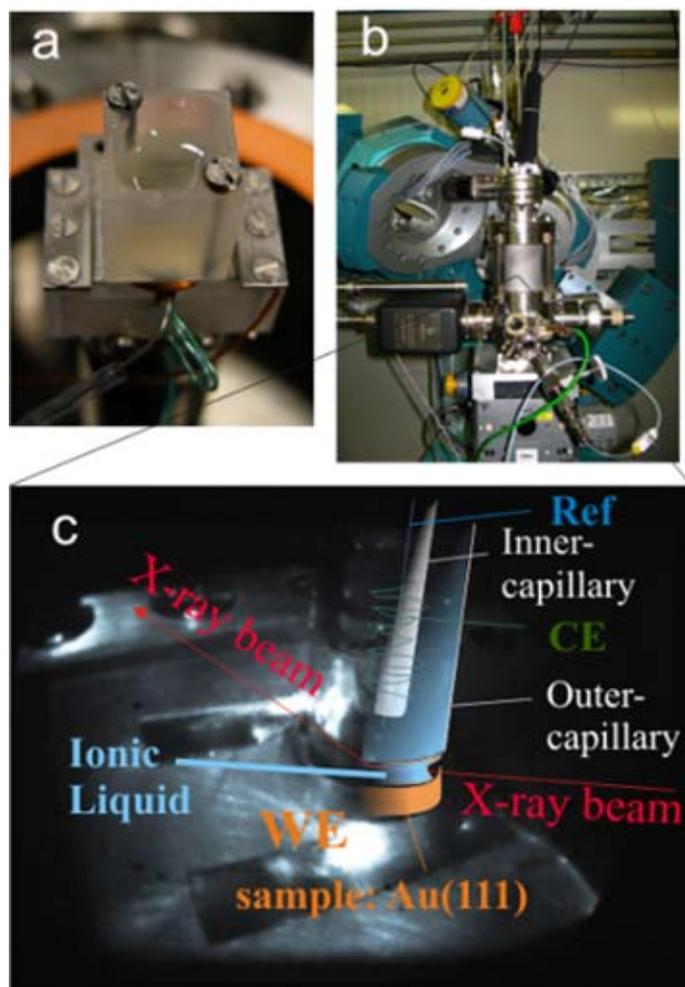
Department of Interface Chemistry and Surface Engineering

Galvanizing is a well known industrial process for protecting steel surfaces, where Zinc coating is carried out by hot dip galvanizing or by electrogalvanizing. The electroplating industry basically uses aqueous solutions due to the high solubility of ions and metal salts resulting in highly conducting solutions. However, usage of the aqueous solution is associated with a drawback that it has a relatively narrow electrochemical potential window, which poses problems during electrodeposition of metals with large negative reduction potentials (e.g. Zn, Al, or Mg). The electrodeposition of Zn carried out in aqueous solution is affected by poor current efficiencies and hydrogen embrittlement of the substrate. For deposition of such metals the aqueous electrolytes might be replaced in the future by non-aqueous electrolytes, which are named as 'ionic liquids' [1-2]. In general terms, ionic liquids (IL) are molten salts with the melting point below 100°C. The advantages of ionic liquids over conventional aqueous electrolytes include a wide electrochemical window and high solubility of metal salts such as ZnCl<sub>2</sub>. One problem usually associated with ionic liquids is their high viscosity and therefore less conductivity in comparison to aqueous electrolytes, which can be addressed by using elevated temperatures. Ionic liquids also have very low vapor pressure, which means they can be handled in UHV environments. The electrodeposition of metals (Zn, Al) using ionic liquids under laboratory conditions and their ex-situ characterization has already been performed [3].

X-ray techniques are an important tool in material science to investigate the crystalline structure of materials. The availability of high brilliance synchrotron X-ray radiation sources has enabled the routine surface crystallography in ultra-high vacuum (UHV) [4-6] as well as in non-UHV [7-11] environment. High energy synchrotron X-rays have been proven useful to probe the crystalline order of the solid-gas [12-14], solid-liquid [7-11], solid-electrolyte [15-17] or electrochemical interfaces [18] as they can penetrate matter with ease and allow *in situ* access to structural parameters. Also, it allows to track the changes occurring at the electrochemical interfaces (surface reconstructions [15], ordered adlayers [16,19], double layer structure, etc) as a function of applied potential, which is hardly possible otherwise (in ex-situ examination). The direct transfer of samples from an electrochemical cell operated in an inert gas

atmosphere into UHV and vice versa proved already useful in the past [20,21]. The combination of such a transfer mechanism with *in-situ* X-ray diffraction offers a unique possibility to address structural knowledge within a number of possible interesting problems, including electrocatalysis or corrosion [21]. With the very low vapor pressure of ionic liquids, electrodeposition from ionic liquids can thus be studied on ultimately well-defined (UHV-prepared) sample surfaces. Recently, a high resolution X-ray reflectivity study revealed that the room temperature ionic liquids (RTIL) show alternate layering of cations and anions over the charged sample (Sapphire) surface [19].

We investigated the electrochemical deposition and re-dissolution of Zn on Au surface by *in-situ* surface X-ray diffraction, in order to understand the processes occurring at the Zn/metal electrochemical interface. An Au single crystal with a polished (111) surface (MaTeck, Germany) was used as substrate for the experiment. For the *in-situ* diffraction studies, we used a portable electrochemical setup (TRECXI) [21] with an addition of a heating stage which allowed to heat the ionic liquid in UHV conditions. With this heating stage, it is also possible to perform electrodeposition at elevated temperatures around 130 °C. The electrochemical cell (EC) is encapsulated in a concentric capillary system, where the reference electrode (Pt wire) is inserted in the inner capillary, while the counter electrode (Pt wire) is wound around the inner capillary (Fig. 1c). With this setup, the electrolyte contact with the sample surface can be attained in the controlled gas atmosphere. Prior to the experiment, the Au (111) surface was prepared by few sputter-anneal cycles in UHV. The surface quality of the sample was examined by Auger spectroscopy and LEED. The electrolyte comprises of ZnCl<sub>2</sub> and 1-Butyl-3-methylimidazolium Chloride ([BMIm]<sup>+</sup> Cl<sup>-</sup>) in 60:40 mol%. This ionic electrolyte is hygroscopic in nature and requires heating prior to the experiment to remove the absorbed water. Ionic liquid was heated in the portable chamber using a PCTFE container (Fig. 1a) for 12 hours in UHV (1×10<sup>-8</sup> mbar) up to 100 °C. The chamber was then filled with inert gas at ambient pressure and the electrochemical (capillary) cell was inserted by opening a UHV valve. The ionic liquid was pumped into the capillaries by using two syringes. The electrochemical cell was then separated from the rest of the chamber.



**Fig. 1:** picture of the electrochemical setup used at the ESRF. a) The container half-filled with ionic liquid during heating. b) The portable EC cell-UHV chamber with the six-circle diffractometer. c) Schematic of the EC-cell explaining the experimental arrangement.

and was evacuated to allow the sample transfer (Au (111) crystal) from the UHV system. After the sample transfer, the portable chamber (TRECXI) was transported to the diffractometer. During the measurements, the portable chamber was again filled with the inert gas at ambient pressure to introduce the EC cell and a droplet of electrolyte at the end of the capillary was then brought into contact with the sample surface. Fig. 1c shows a schematic of the electrochemical setup. This setup allows tracking the interface changes by *in-situ* X-ray diffraction without exposing the ionic liquid electrolyte and sample surface to ambient air.

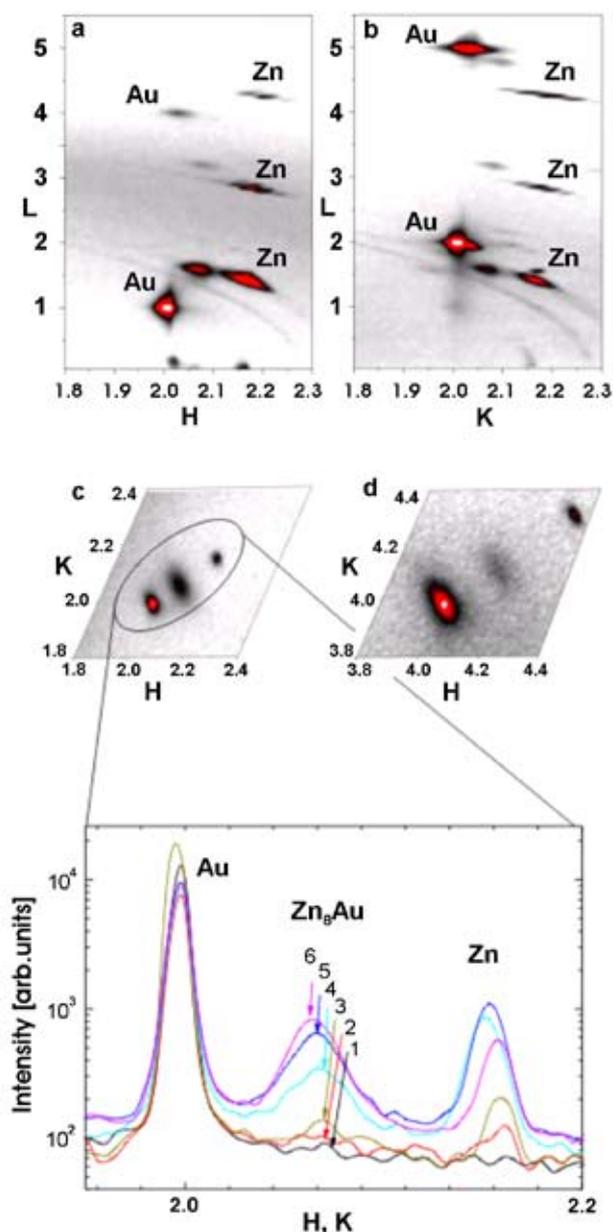
The *in-situ* X-ray diffraction experiments were performed at the undulator beamline ID32 at the European Synchrotron Radiation Facility (ESRF) in Grenoble, France, using a wavelength ( $\lambda$ ) of 0.539062 Å. The incident angle was chosen to 0.2°. The scattering vector  $\mathbf{q}$  is defined as  $\mathbf{q} = \mathbf{k}_f - \mathbf{k}_i$ , where  $\mathbf{k}_i$  and  $\mathbf{k}_f$  are the incoming and diffracted wave vectors, respectively. Au crystal possess a FCC unit cell (lattice parameters  $a_0 = 4.08$  Å,  $\alpha = \beta = \gamma = 90^\circ$ ), which can be transformed to a surface unit cell for

Au (111) surface ( $\mathbf{a}_1 = \mathbf{a}_2 = \sqrt{2} \times a_0 = 5.767$  Å,  $\mathbf{a}_3 = \sqrt{3} \times a_0 = 7.064$  Å,  $\alpha = \beta = 90^\circ$ ,  $\gamma = 120^\circ$ ). This unit cell leads to a hexagonal reciprocal unit cell with lattice parameters  $\mathbf{a}_1^* = \mathbf{a}_2^* = 1.258$  Å,  $\mathbf{a}_3^* = 0.8895$  Å,  $\alpha = \beta = 90^\circ$ ,  $\gamma = 60^\circ$ . To describe the scattering vector  $\mathbf{q}$  and the reciprocal space of Au (111), we used the above mentioned reciprocal unit cell. The in-plane miller indices are then denoted by H & K, while L is the out-of plane index.

The electrodeposition was performed at the potential of -1.5 V. Fig. 2 shows the first results of *in-situ* X-ray diffraction on Au (111) with Zn containing ionic liquid electrolyte. The sequence of in-plane scans measured in (1, 1) direction (with respect to time), after applying the potential are shown in Fig. 2. The in-plane scan measured directly after applying the potential (scan no. 1), shows only one reflection at H = K = 2, which corresponds to the Bragg reflection (2, 2, 0.05) of the Au substrate. With time (scan no. 2), additional in-plane intensity was observed at (2.16, 2.16, 0.05), which grows further (scans no. 2-5). The d-spacing for this reflection was calculated as 1.33 Å, which corresponds to the (11-20) Bragg reflection of Zinc. Furthermore, other than the pure Zn reflection, a new in-plane reflection developing at (2.06, 2.06, 0.05) was also observed, can be explained by the formation of AuZn<sub>8</sub> alloy. These reflections show that the Zn and Zn-Au alloy are epitaxially ordered with respect to the Au (111) substrate. The Zn and Zn-Au alloy lattices are hexagonal closed packed lattice. Following the Zn-Au phase diagram [22], for lower Zinc concentrations the alloys possess cubic, tetragonal or orthorhombic structures ( $\alpha_{1-2}$ ,  $\alpha'_{1-2}$ ,  $\beta$ ,  $\beta_1$ ,  $\gamma$ ,  $\gamma_2$ ,  $\delta$ ), while for the Zn concentration greater than 75 at.% the alloys have hexagonal or hcp structure ( $\gamma_3$ ,  $\epsilon$ ).

Fig. 2a-d shows the out-of-plane and in-plane maps of the Au surface with Zn deposition, respectively. The out-of-plane maps (Fig. 2a & 2b) show the Zn and Zn-Au alloy peaks along with the Au substrate peak. The peaks are epitaxial and symmetrical arranged in reciprocal space. From the in-plane maps in (1, 1) direction, the gold and Zn peaks are clearly seen (AuZn<sub>8</sub> and Zn<11-20> || Au<220>).

We also observed the formed pure Zn peak to get smaller with time, as can be seen from in-plane scan (scan no. 6) in Fig. 2. Here, the reflection corresponding to the AuZn<sub>8</sub> alloy intense further, while intensity of the reflection corresponding to the pure Zn phase reduces. This observation indicates the pure Zn phase dissolves or transforms in an Au-Zn alloy; however, more experiments are required to corroborate this behavior.



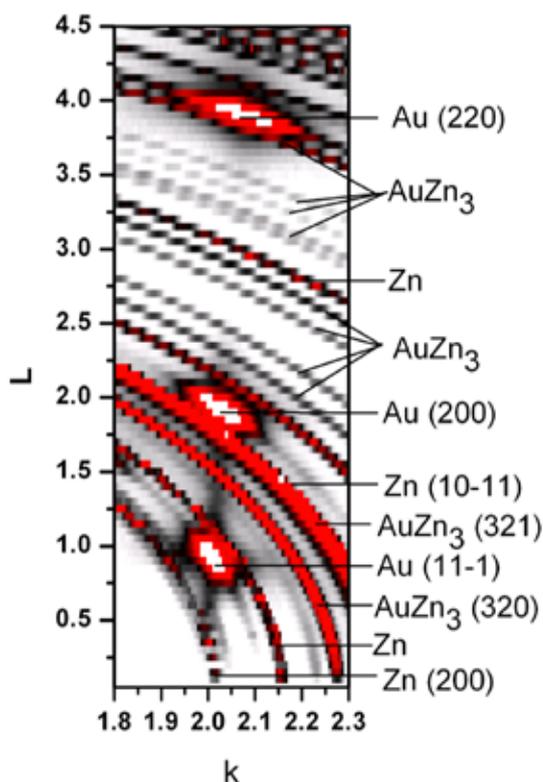
**Fig. 2:** Data observed from the *in-situ* X-ray measurements. a) & b) Out-of-plane reciprocal space maps showing the Au substrate peak at (201), (022) and epitaxially ordered Zn and AuZn<sub>3</sub> peaks along with their symmetrical equivalents. c) & d) In-plane maps in (1, 1) direction showing the three peaks of Au, AuZn<sub>3</sub>, Zn peaks. Several in-plane scans measured with increase in time showing the intensity evolution of various peaks.

To compare the *in-situ* X-ray diffraction data obtained from the electrodeposition on the Au (111) surface, we performed the ex-situ X-ray diffraction on the Zn-Au film electrodeposited on the textured Au (111) sample. In this case, the textured Au (111) polycrystalline film was deposited by PVD on the glass substrate. The electrodeposition was performed in glove box (H<sub>2</sub>O, O<sub>2</sub> < 2 ppm) using the conventional three electrode electrochemical cell with Pt wire as reference and counter electrode, at the potential of -1.5V for 2 hrs. After the experiment, the excess electrolyte was rinsed from the sample using ethanol and the sample was dried in N<sub>2</sub> stream.

The surface X-ray diffraction was performed with the same wavelength and incidence angle as mentioned above. The sample was mounted on a Teflon holder covered with Kapton foil to isolate the sample from the atmosphere. A stream of N<sub>2</sub> was used to rinse the closed assembly during the whole measurement.

Fig. 3 shows the out-of-plane reciprocal map for Zn/ZnAu-Au-glass sample. From the map, Au substrate peaks can be seen along with several rings for the Zn/Au-Zn alloy phases. Also, it is evident that reflection from the Zn-Au alloy phase (AuZn<sub>3</sub>) is most intense in the Zn-Au coating. Furthermore, despite the fact that the substrate has preferential (111) orientation, the formed coating (Zn/AuZn<sub>3</sub>) is polycrystalline. The reflections from the pure Zinc phase have very low intensity, which indicates that the ex-situ Zn electrodeposition resulted mainly in the formation of a AuZn<sub>3</sub> alloy.

The presented preliminary results of *in-situ* and ex-situ X-ray diffraction experiments suggest that during electrodeposition, a pure Zn film deposits first; but, as time elapses, an additional formation of a Zn-Au alloy is observed. However, further experiments are required to obtain conclusive mechanisms. In future, factors such as the influence of the respective potential sequence in time, the surface roughness, the presence of residual water/oxygen in the ionic electrolyte will be further studied. Also, these experiments will be extended to more practical systems like Fe/Zn or Fe/Al.



**Fig. 3:** Out-of-plane map for the Au-Zn alloy film on the textured Au (111) film on the glass substrate.



In conclusion, we observed the epitaxial growth of hexagonal Zn and AuZn<sub>8</sub> on clean Au (111) surfaces, with the orientation Zn<11-20> || Au<220>. The current investigation proves that the processes occurring at Au/Zn-IL electrochemical interfaces can be successfully probed to obtain detailed structural information during deposition.

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# Deformation Laminate Microstructures in Shear-Deformed Copper Single Crystals

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Many studies on the behavior of metals under stress aim at understanding the evolution of plastic deformation. Since plastic behavior of materials is the result of the interaction of lattice defects at several length scales, the overall macroscopic properties are strongly influenced by the formation of internal microstructures. These internal structures generally arise when non-homogeneous deformation of a material is energetically favorable compared to a uniform state. The main concern of this project is to obtain a better understanding of deformation patterning phenomena in plasticity, particularly regarding the formation of deformation laminates. A formation of laminate microstructures is associated with the activation of single-slip deformation regions in neighboring zones of the same portion of material involving different slip systems [1,2].

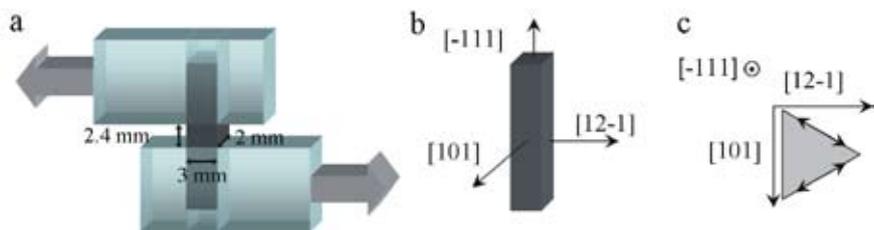
In this work, we study the formation of lamination microstructures in plastically-deformed copper single crystals under well-defined single-pass loading conditions. In simple shear experiments, nearly uniform macroscopic strain could be attained with the use of a sample geometry in which friction effects are negligible in the region of interest [3]. The evolution and spatial distribution of the deformation was traced on the surface of the sample using digital image correlation (DIC). The microstructure of the sheared material was then characterized using orientation microscopy based on the analysis of the electron back-scattered diffraction pattern (EBSD). The copper single crystals (99.98% purity) were cut by spark erosion. The shear tests were performed on a miniaturized device using a tool displacement rate of 5 μm/s, Fig. 1a.

Fig. 1b shows the initial orientation of the undeformed single crystal (101)[12-1]. The shear load is applied along the [12-1] direction, Fig. 1b. Under these loading conditions, two coplanar slip

systems have Schmid factors of approximately 0.63 (A2 and A6 in Table 1). These two slip systems are shown schematically in Fig. 1c. The gray-colored triangle corresponds to their common {111} plane which is oriented parallel to the load direction. Since the primary active systems are coplanar, one can consider their sum as a single system for which the activation direction is the direction of the applied load.

In order to measure the strain on the surface of the specimen in-situ, we applied the digital image correlation method (DIC) [5,6] which is based on the recognition of geometrical changes in the gray scale distribution of surface patterns after straining. The structural characterization of the surface of the samples and the orientation microscopy were performed in the scanning electron microscope (SEM) Jeol JSM 6500F with a field emission gun operated at 15 kV. The electron back scattered diffraction pattern were recorded and evaluated by the EDAX/TSL EBSD System equipped with a Digiview camera.

The single crystal was deformed in simple shear to  $\gamma = 0.2$ . The load/displacement curve was recorded during the shear deformation at a step size of



**Fig. 1:** a: Schematic drawing of the specimen holder with the specimen used for the shear experiments; b: Crystallographic orientation of the single crystal sample positioned in the sample holder; c: Schematic orientation of the primary active slip plane (gray triangle) according to the given crystallographic orientation. The individual slip directions A2 and A6 are indicated by the arrows.

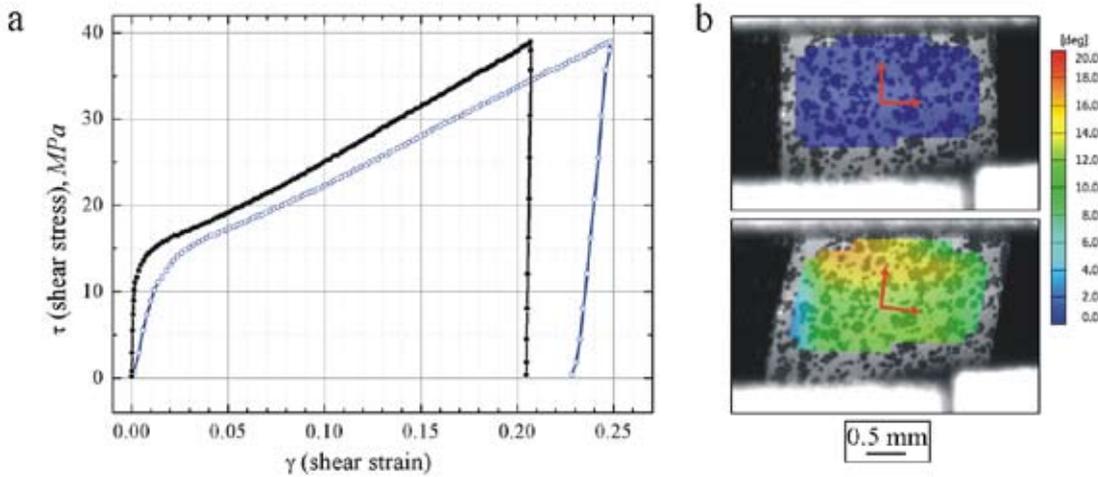
5 μm. Fig. 2a (next page) presents the dependence between the applied shear stress  $\tau$  and the shear strain  $\gamma$  as calculated from the load/displacement data (blue curve). Via the DIC approach, digital images of the specimen surfaces were recorded during the

B2	B4	B5	A3	A2	A6	C1	C3	C5	D4	D1	D6
$\pm[011]$ (111)	$\pm[101]$ (111)	$\pm[110]$ (111)	$\pm[101]$ ( $\bar{1}11$ )	$\pm[011]$ ( $\bar{1}11$ )	$\pm[110]$ ( $\bar{1}11$ )	$\pm[011]$ ( $\bar{1}\bar{1}1$ )	$\pm[101]$ ( $\bar{1}\bar{1}1$ )	$\pm[110]$ ( $\bar{1}\bar{1}1$ )	$\pm[101]$ ( $\bar{1}\bar{1}1$ )	$\pm[011]$ ( $\bar{1}\bar{1}1$ )	$\pm[110]$ ( $\bar{1}\bar{1}1$ )

**Tab. 1:** Schmid and Boas's FCC slip system nomenclature taken from [4].

S  
E  
L  
E  
C  
T  
E  
D

H  
I  
G  
H  
L  
I  
G  
H  
T  
S



**Fig. 2:** a: Dependence between the shear stress and shear strain obtained from the load/displacement measurement (blue curve) and from the DIC method (black curve); b: Digital images of the sample surface decorated for the DIC method before (upper image) and after the deformation (lower image) overlaid with the obtained distribution of the shear angles according to the color scale.

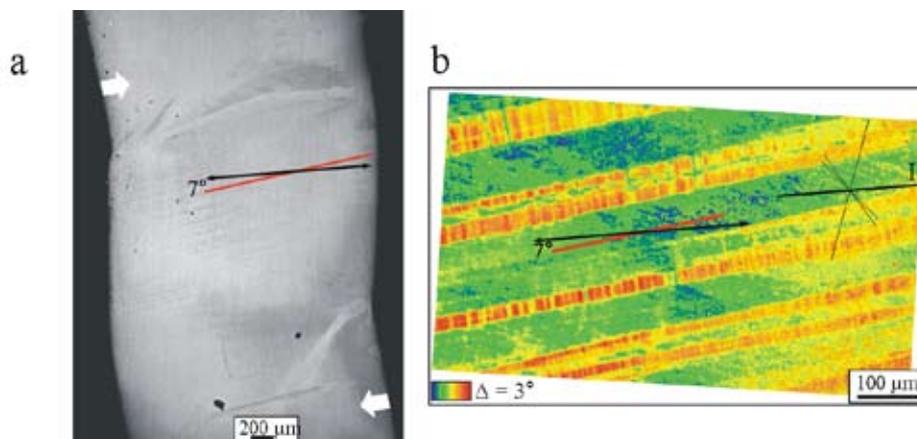
experiment, Fig. 2b. The images were overlaid with the obtained strain fields that reveal the distribution of the shear angles on the specimen surface. Fig 2b shows an inhomogeneous deformation with some concentration of strain near the clamps. There is a larger region of homogeneous deformation in the central area of the sample. The change of the shear strain in this region measured by DIC in dependence on the applied load is then displayed by the black curve in Fig. 2a.

The strain measurement also reveals the distribution of the macroscopic strain and of the deformation gradient  $F$  on the surface of the deformed crystal.

In order to analyze the deformation in more detail, we compare the experimentally observed deformation gradient  $F$  to a simple shear situation  $G(\gamma, \varphi)$  which can be characterized by two parameters, namely, the magnitude of the shear  $\gamma$  and the orientation of

the shear normal with respect to the vertical axis (expressed by the angle  $\varphi$ ). The comparison is done by minimizing an energy function  $E(F, G)$  that measures the difference between a given strain  $G$  and the observed deformation gradient  $F$ . Averaging the deformation gradient in the homogeneously deformed sample region yields after energy minimizing a simple shear  $G$  of magnitude  $\gamma = 0.20$  which conforms to the observed final shear strain. However, in the analysis the normal of the shear is rotated clockwise by an angle of  $\varphi = 4.5^\circ$  from the vertical direction which indicates that the effective direction of the shear load differs from its initial direction.

Fig. 3a shows a back scatter electron (BSE) micrograph of the single crystal after shear deformation. The direction of the applied shear load is indicated by arrows. The formation of glide bands can be observed. The direction of the bands is indicated by the red line, and the microbands of the deformed



**Fig. 3:** a: Back scatter electron optical overview micrograph of the copper single crystal after shear deformation. The direction of the applied load is shown by the white arrows; b: EBSD map of an area in the deformed part of the specimen which shows the relative crystallographic orientation changes within  $3^\circ$  according to the color scale. The plane traces of the slip planes calculated from this orientation map are presented in the right corner. The black arrows represent the trace of the primary slip plane. The direction of the microbands which are tilted to  $7^\circ$  with respect to the slip plane is signified by red lines (in a and b).



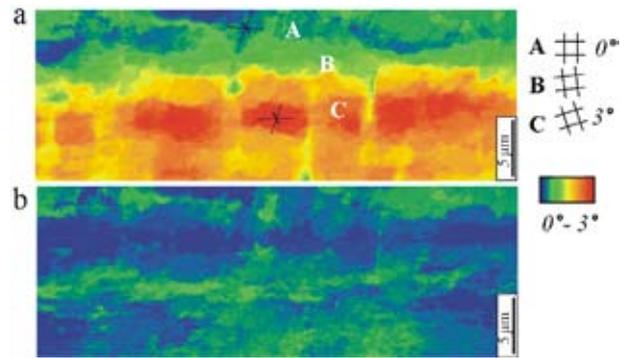
single crystal are oriented parallel to the initial position of the crystallographic plane of the primary slip, i.e., they are aligned along the direction of the applied shear load.

Fig. 3b shows the EBSD map of the deformed part of the crystal (step size  $2\ \mu\text{m}$ ). It shows the relative variations in the crystal orientation within  $3^\circ$ . The formation of the microbands with a different orientation (red) compared to the material in between (green/blue) can be observed from this map. The plane traces of the  $\{111\}$  slip planes are displayed according to the averaged lattice orientation of the investigated area, Fig. 3b. As shown in Fig. 1c, the common slip plane of the primary slip systems is initially oriented parallel to the shear load (along  $[12-1]$ ) and is shown in Fig. 3b by a black line. When comparing the direction of this  $\{111\}$  plane with the direction of the microbands (displayed as a red line), a deviation of about  $7^\circ$  can be observed. Thus, the direction of the microbands is not crystallographic. Furthermore, since the development of the microbands occurs along the applied load and parallel to the initial position of the primary slip plane, a macroscopic tilt of the crystal lattice out of the initial state to about  $7^\circ$  can be determined in the final state of deformation. A further feature of the bands is the additional patterning inside of the “red” microbands which are subdivided into parts by orthogonal intersections.

Fig. 4a shows the high resolution EBSD measurement of the area that includes the microband and the surrounding material (step size  $0.1\ \mu\text{m}$ ). The EBSD map presents the orientation distribution relative to a reference point (initial orientation). The red-colored zones in Fig. 4a show the area on the edge of the microband. The green/blue areas correspond to the material outside of the microband. The orientation difference indicates that the crystal lattice inside of the band is tilted by  $3^\circ$  in comparison to the material outside of the band.

In Fig. 4b, the variation of the crystal orientation out of the normal direction is displayed in the same orientation deviation range as in Fig. 4a. However, Fig. 4b reveals no significant rotation of the lattice out of the front face. This observation shows that the lattice rotation of approximately  $3^\circ$  occurs only within the plane as schematically shown on the right hand side in Fig. 4a. The rotation of the lattice inside of the band occurs counterclockwise compared to the outside region.

In order to better understand how and why the material forms the observed microstructure patterns, we reconstruct the observed microstructure by a lamination model assuming infinite cross-hardening.



**Fig. 4:** a: High resolution EBSD map on the edge of a microband. The colors indicate the relative orientation changes of the lattice within  $3^\circ$ . The plane traces of the slip planes are presented for the areas inside (red) and outside (green/blue) of the microband. Graphical illustration of the observed local lattice rotation is presented on the right hand side; b: High resolution EBSD map on the same area shows the variation of the crystal orientation out of the normal of the image plane according to the color scale.

A certain type of kinematically compatible microstructures consists of so-called rank-one laminates. If one assumes that the deformation is piecewise affine and that there are only two affine components,  $F_1$  and  $F_2$ , then the requirement of kinematic compatibility is equivalent to the existence of an invariant plane, i.e., a plane that is stretched equally by both  $F_1$  and  $F_2$ . The two differently deformed material domains can then be “glued” together on this invariant plane, the normal of which is the lamination direction [7,8].

We consider the orientation of the sample during the shear experiment as illustrated in Fig. 1. A completely homogeneous shear of this type can be accommodated by an activity in the A2 and A6 slip systems (Table 1). Due to the free boundary conditions, a variety of possibilities exist to satisfy this deformation in a kinematically compatible manner. The DIC-measurement, however, reveals that the actual deformation differs somewhat from this simplified assumption. Instead, there is a large, fairly homogeneous region with average strain  $U$  in the final deformation state.

An analysis was conducted in order to identify a deformation that contains two compatible deformation gradients  $F_1$  and  $F_2$  which yield the macroscopic strain obtained from the measurement. The analysis uses a gradient flow method for the magnitudes of slip in the involved slip systems with respect to the deformation energy. The results show that the major plastic deformation component  $F_1$  implies an equal activity in the primary slip systems A2 and A6. For the secondary component of the plastic deformation,  $F_2$ , there are several different choices that are compatible with the major deformation. Two possibilities stick out: equal mixture of the C1 and C5 as well as of the B4 and D4 slip systems are both compatible regardless



of the magnitude of slip. The combination of slip on the systems B4 and D4 together with primary slip on systems A2 and A6 yields a lamination structure as observed in the experiment. This means that first the lamination direction is in this analysis rotated approximately  $7^\circ$  counterclockwise with respect to the  $[-111]$  direction as found in the experiment (Fig. 3b). Second, the lattice in that area where systems B4 and D4 are active (red regions in Figs. 3b and 4a) is rotated counterclockwise by approximately  $3^\circ$  in comparison to the neighbouring area with the primary active A2 and A6 systems. Therefore, a theoretical analysis assuming infinite cross-hardening correctly predicts a lamination microstructure in the shear-deformed copper single crystal that corresponds almost exactly to the orientation patterning observed in the EBSD-measurement.

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## Application of SKP for *in-situ* Monitoring of Ion Migration along Insulator/Insulator Interfaces

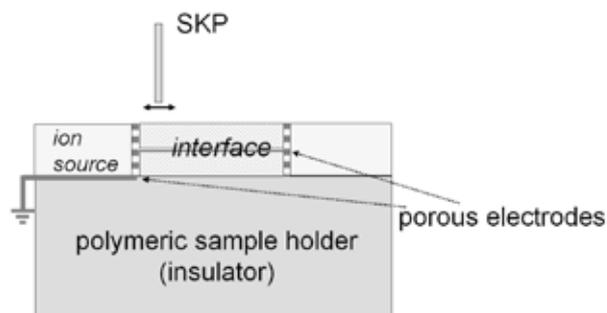
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Department of Interface Chemistry and Surface Engineering

Since its first application in corrosion science [1] Scanning Kelvin Probe (SKP) has proven to be a unique and very useful tool for studying delamination at polymer/metal interfaces and over the last 20 years has experienced a steadily increasing popularity for corresponding studies. What is not that widely known is that in the absence of oxygen SKP can also be applied for studying diffusion/migration of ions along polymer/metal interfaces, as was shown by Stratmann et al. already as early as 1992 [2].

Diffusion/migration of ions plays an important role in many technical processes. For instance, we could show that the performance of conducting polymers for corrosion protection critically depends on the transport of anions with respect to the one of cations within the coating [3]. It could be shown in this work that over the distances relevant for realistic corrosion scenarios all conducting polymers will finally show cation permselectivity, which will lead to sudden break down of corrosion protection and even enhancement of corrosion. This can be prevented by avoiding macroscopic percolation networks of conducting polymer in the coating. Then excellent performance can be achieved [3,4]. Why this is the case could only be unravelled by fundamental cutting edge research applying the SKP for studying the ionic transport properties of conducting polymers over extended distances [3].

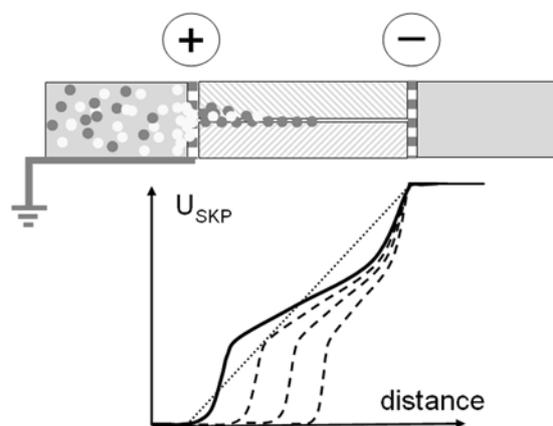
Now we found that SKP can be applied not only for studying ionic transport in conducting polymers or along polymer/metal interfaces. Surprisingly, no conductive substrate is necessary, i.e. the ion diffusion/migration along any insulator/insulator interfaces should be possible to study by SKP. The idea is shown in Fig. 1. The insulator/insulator



**Fig. 1:** Experimental set-up for the measurement of ion mobility along interfaces. The sample with the interface of interest is placed on a thick insulator and between two electrodes over which a bias is applied. At least on one side a reservoir of ions, e.g. an aqueous electrolyte, is required as ion source.

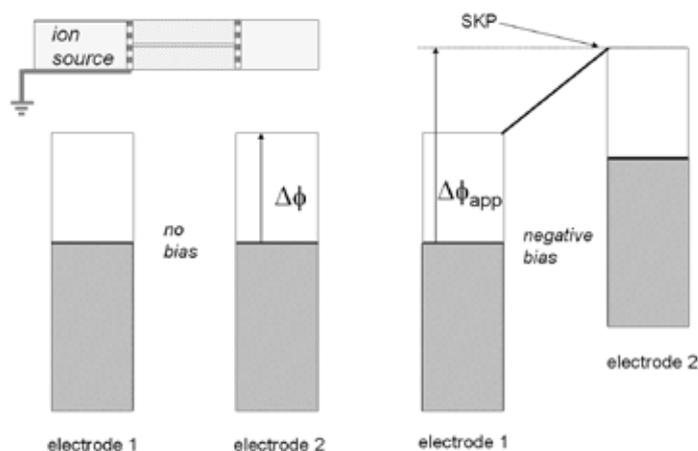
interface is placed laterally between two electrodes, one of them connected to the Kelvin probe, usually via ground. This electrode is in contact with a reservoir of ions and a bias is applied between the two electrodes. As will be discussed further below, this is required for monitoring the ion movement along the interface. In order to ensure a potential gradient across the full extension of the sample the interface has to be placed far above the grounded base plate of the measurement chamber of the SKP. This is achieved by using a sufficiently thick plastic sample holder which effectively prevents any significant capacitive effects between the electrodes and the metallic base plate.

Fig. 2 shows what will happen, if ions move into the interface while a bias is applied over the two electrodes. The full line sketches the starting situation. Since the two electrodes are limited in size the field of them is not linear, as it should be inside an ideal plate



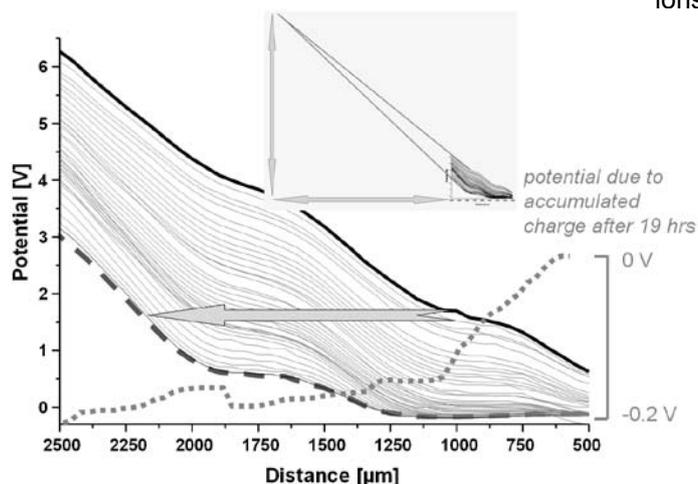
**Fig. 2:** Schematic sketch of the ion progress along the interface and the resulting distortion of the potential profile along the interface (mark: due to the thinness of the sample in reality the electrodes are larger and protrude vertically, providing a hindrance for SKP scanning). Dotted: potential distribution for the case of an ideal plate condenser geometry; solid line: potential for a more realistic starting situation; dashed line: evolution during progress of ions.

capacitor (dotted line), but stronger in the vicinity of the electrodes and weaker in the middle between them. As ions move into the interface the Ohmic resistance is drastically lowered and the potential is pulled towards the one of the electrode, causing a potential shift as sketched out in the sequence of dashed curves. It is important to notice that in this experimental set-up the potential measured by SKP is



**Fig. 3:** Left: Energy levels of the two electrodes on both sides of the sample without applied bias. Over electrode 2 the SKP will measure the correct work function  $\Delta\phi$ . Right: when electrode 2 is negatively biased versus electrode 1, its electronic energy levels shift upwards, resulting in a seemingly higher work function  $\Delta\phi_{app}$ , because the grounded electrode 1 is the reference point.

an apparent potential bearing the wrong sign. This is explained in Fig. 3. If both electrodes are comparable and no bias is applied, the starting situation is as shown on the left hand side of Fig. 3. No potential gradient would be measurable along the scan over the sample. If a potential is applied, the energy levels of the two electrodes are shifted with respect to each other. In the figure electrode 2 is biased negatively over electrode 1. The Scanning Kelvin Probe senses the local vacuum level at each position of the scan across the sample [5]. Over electrode 2 it senses the vacuum level with reference to electrode 1, which is grounded and hence the reference point for the SKP. Hence, electrode 2 appears to have a much



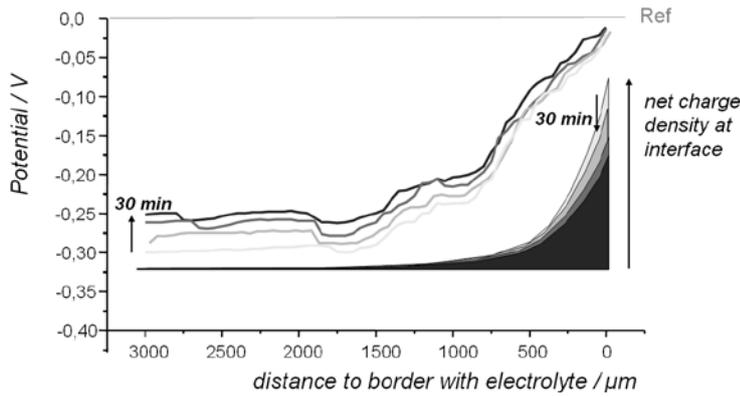
**Fig. 4:** Potential curves obtained over an epoxy/glass interface. A glass plate modified by a monolayer of silanes and coated with a layer of epoxy paint. The sample was placed between two mesh electrodes, with the electrolyte containing sodium and chloride ions on the right hand side. The sample size was about 10 mm between the electrodes and the applied bias was 22 V (thick solid line: first measurement; dashed line: last measurement, obtained after 19 hrs; dotted line: potential profile obtained after 19 hrs and switch-off of applied bias).

higher work function than it really has, higher by exactly the applied bias between the two electrodes. Thusly, when electrode 2 is biased negatively against electrode 1 the measured potential has the opposite sign.

Of course, the evolution of the potential curves measured by SKP while ions move into the interface is directly correlated to the mobility of these along the interface. Their progress is described by the Nernst-Einstein equation and contains a diffusion term and a migration term. As the potential gradient is directly adjustable, the direct approach to facilitate the situation would be to increase it to values where the mobility is mainly determined by migration, thus bringing us a step forward to obtaining the correlated mobility coefficients.

In Fig. 4 one of our first measurements on an insulator/insulator sample is presented. A glass plate was modified by a monolayer of silanes and coated with a layer of epoxy paint. The sample was placed between two mesh electrodes, just as sketched in Fig. 1, with the electrolyte containing sodium and chloride ions on the right hand side. The sample size was about 10 mm between the electrodes and the applied bias was 22 V. This is a relatively low potential gradient, however, at the moment our SKP set-up has a maximum potential range of about 6 V. This is why higher biases were not applied yet and also why the sample could not scanned across its full extension (see insert in Fig. 4). As can be seen, the measured curves change similarly to what has been proposed in Fig. 2. Hence, it is indeed possible to follow the incorporation of ions into an insulator/insulator interface. Now, if the mobility were mainly driven by migration, then from these curves one could try to directly obtain the mobility coefficients.

However, for this also the concentrations of species are required. Although these are not directly obtainable, at least the incorporated net charge can be directly obtained, just by switching off the applied bias. Then all deviation measured by SKP from the potential of the right hand electrode will be correlated to net charge accumulated at the interface. This is shown by the dotted curve, obtained after 19 hrs of measurement and then switching off of the applied bias. As can be seen the potential measured by SKP shows a well discernible profile. The potential profile seen here is caused by an excess of positive charge accumulated at the interface. The charge neutrality is assumed to be kept by a corresponding negative excess charge near the electrode which cannot be measured by the SKP due to a necessary minimum distance that has to be kept to the



**Fig. 5:** Potential profiles across the interface during decay of accumulated charge.

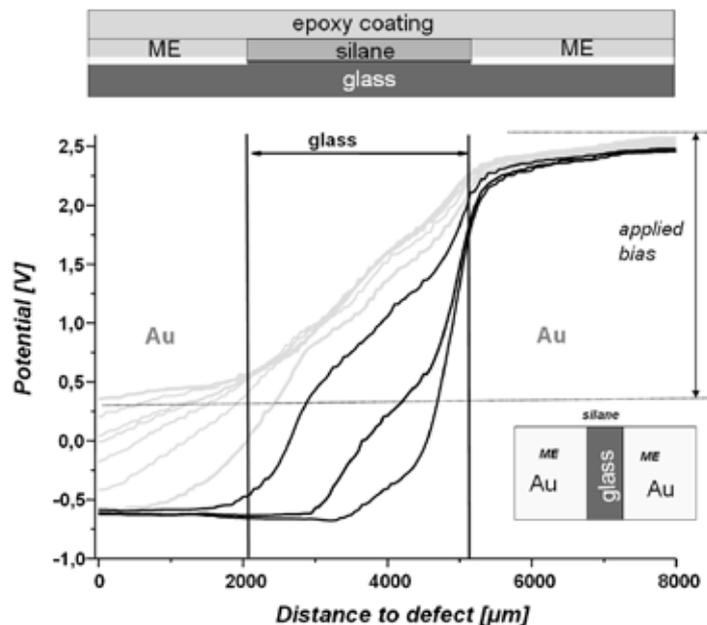
mesh electrode. Principally, the charge distribution at the interface can be derived from the measured potential profile. This is subject of ongoing research. If due to high biases only either cations or anions are driven into the interface, then the measured charge distribution is directly providing the distribution of concentration of the respective ions. Then it should be possible to obtain the mobility coefficients from such measurements.

In absence of an external bias this accumulated charge dissipates, which again can be directly followed by SKP. An example is shown in Fig. 5, where a similar experiment was performed and the decay of the potential profile was recorded for 30 minutes.

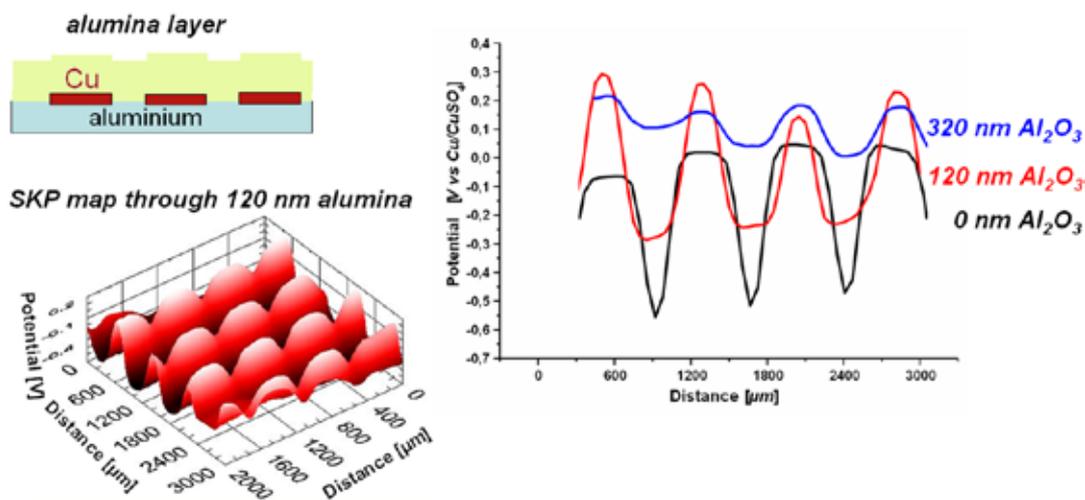
In order to allow positioning of the SKP tip directly over the edge between electrode and insulator/insulator interface we designed a modified set-up. This is shown in Fig. 6. Gold electrodes were directly evaporated onto the glass substrate, as stripes on the left and right hand side of the sample. Again a bias is applied between the two electrodes. The gold surface was modified with mercaptoethanol (ME) which ensures a weak interface between epoxy and the gold surface. The glass surface was again modified by silane, which ensures a relatively stable interface between glass and epoxy. Then the epoxy coating was applied and on the left hand side the sample was brought into contact with the electrolyte and the gold edge exposed there polarized to a negative potential of  $-700 \text{ mV}_{\text{SHE}}$  (details of such a set-up are described e.g. in [3]), where the ME modified, epoxy coated gold surface is known to be quickly delaminated (grey curves in Fig. 6). This results in a thin layer of electrolyte between the gold and the epoxy coating reaching quickly the edge between the gold layer and the glass. This

is the point where the incorporation of the ions into the epoxy/glass interface starts (black curves). Much better than can be seen in Fig. 4 the potential curves shown in Fig. 6 follow the predicted behaviour sketched in Fig. 2. Here the full potential profile between the two electrodes can be seen, including the two borders between glass and gold. As discussed above the potential profile measured by SKP across the insulator part of the sample shows the wrong sign, i.e. the right hand gold electrode is biased negatively against the left hand electrode. The potential change on the left gold electrode due to the delamination, however, shows the correct sign, as it is directly measured over the grounded gold electrode.

These examples just give a first idea of how SKP can be applied for studying mobility along any insulator/insulator interface of interest. This technique should also be applicable for ceramic/ceramic interfaces (as well as to metal/ceramic interfaces). In how far the interface is accessible to SKP depends on the electronic charge mobility in the ceramic. Assuming electronic equilibrium at the interface a band bending correlated to the corresponding charging has to be expected. As the electronic charge density is low only very thick layers of insulator are expected to show their own work function at the outer surface. For thinner layers the band structure of the



**Fig. 6:** Potential curves obtained over an epoxy/glass interface obtained in a modified set-up where the electrodes are not placed vertically against the sample edges, but where they are directly placed on the glass substrates (as stripes, left and right) and are also directly coated by the epoxy coat. In order to weaken the gold/epoxy interface for a faster start of the measurement of diffusion/migration along the glass/epoxy interface the gold electrodes have been modified by mercaptoethanol (ME). This set-up allows scanning also over the electrodes.



**Fig. 7:** Upper left: schematic sketch of the pattern of copper ( $100 \times 100 \mu\text{m}^2$  squares, separated  $100 \mu\text{m}$  from each other) on aluminium, with additional alumina coating on top. Bottom left: resulting SKP map over uncoated sample. Right: Potential profiles across a row of copper squares on aluminium, uncovered (black line), covered by  $120 \text{ nm}$  alumina (red line) and covered by  $320 \text{ nm}$  (blue line).

whole layer will be affected by the underlying material, i.e. information about the interface is obtained at the surface of the outer layer.

In Fig. 7, a pattern of copper squares was evaporated onto aluminium and subsequently alumina was evaporated to form layers of different thickness. As can be seen the potential contrast that is measured by SKP sensitively depends on alumina layer thickness. However, even up to a thickness of  $320 \text{ nm}$  the copper squares still can be seen beneath the alumina layer.

In summary, we have shown that SKP is not only applicable to measuring diffusion and migration of ions along interface between polymers and metals, but also to interfaces with non-conductive substrate. For monitoring the advancement of the ions along the interface a bias has to be applied across the sample. As mobility of charge carriers plays an important

role in many applications, e.g. in fuel cells or supercapacitors, these findings open a completely new and wide field for the application of SKP.

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## Towards *in-situ* Spectroscopic Detection of Ions under Polymer Films: Water and Ion Orientation near Semiconductor Interfaces

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Organic coatings have a variety of applications not only in corrosion protection, but also in a variety of other fields, including the microelectronics industry. The mechanisms leading to a failure of these coatings has been studied for a number of years [1,2]. This includes the investigation of the diffusion and migration of water and salt solutions along buried interfaces. *In-situ* experiments using the scanning Kelvin probe yield complex signals which involve contributions from water and ions. The element distribution along polymer/solid interfaces has been investigated *ex-situ* by removing the polymer coating from the metal surface and investigating the solid surface by x-ray photoelectron spectroscopy under ultra-high vacuum conditions [1,2].

Here, we report first steps towards *in-situ* experiments characterizing both water and ion diffusion along interfaces at the same time. Infrared (IR) spectroscopy has in the past been used to study the diffusion of water under and through polymers [3,4]. Because there are several ions absorbing infrared radiation, IR spectroscopy is suitable to study the lateral distribution of ions as well.

The use of metal substrates is ideal for studies comparable to „real-world“ technical systems. It does, however, bring problems in optical, especially infrared, experiments near an interface. Ideal substrates are transparent materials. Easily available semiconductor materials such as silicon and germanium are transparent in the mid-infrared wavelength range. Furthermore, doped semiconductors with specified conductivities are available, enabling concurrent electrochemical and optical experiments.

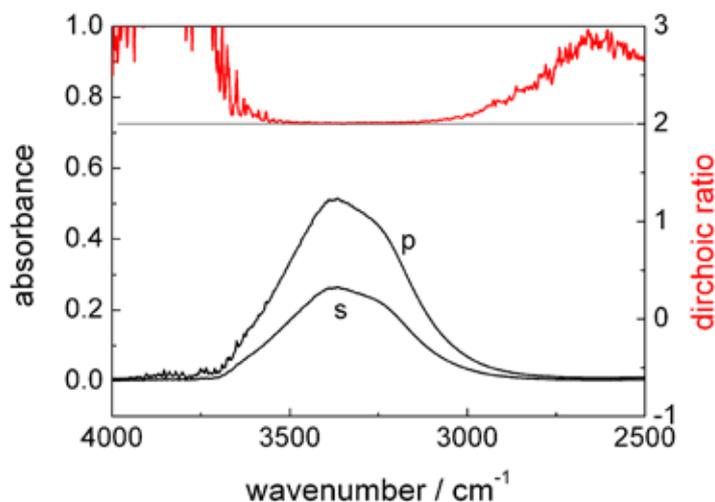
Their transparency makes Si and Ge ideal substrates for use in attenuated total reflection infrared (ATR-IR) spectroscopy, where a “near surface region” is probed by an with distance from the interface exponentially decaying evanescent wave. The decay length of the electric field profile under the conditions used here is  $\sim 300$  nm. In addition to just confirming the presence of structures near a surface, the use of polarized ATR-IR spectroscopy offers insights into the orientation of molecules and ions near a surface [5].

As a first step towards the understanding of the complex interactions between water and ions underneath polymer films, we report results from studies of their orientation near interfaces between Si(111) substrates with their native oxide layer and aqueous solutions of different salts. The salts used here are  $(\text{NH}_4)_2\text{SO}_4$ ,  $\text{NH}_4\text{Cl}$ , and  $\text{NH}_4\text{NO}_3$ , containing the infrared-active cation  $\text{NH}_4^+$  and infrared-active ( $\text{SO}_4^{2-}$ ,  $\text{NO}_3^-$ ) as well as inactive ( $\text{Cl}^-$ ) anions.

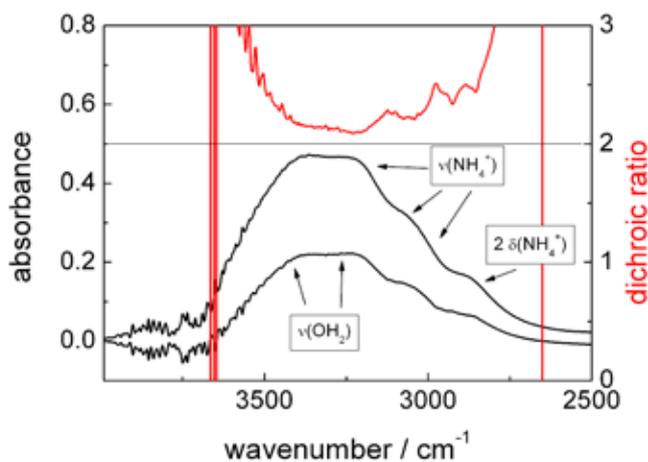
Information about the orientation of molecules is obtained through the dichroic ratio  $R$ , i.e. the ratio of absorbance measured with  $p$  and  $s$  polarisations. At an angle of incidence of 45 degrees, as used here, a ratio of 2 corresponds to an isotropic orientation of the molecules [5].

Fig. 1 shows ATR-IR spectra with  $s$  and  $p$  polarisations for pure water, as well as the plot of the ratio of these two spectra in the region of the  $\text{OH}_2$  stretching modes. The straight line observed at a dichroic ratio of them confirms the average isotropic orientation of the water in the near-surface region as probed here.

In the presence of salts, the behaviour may change, as shown in Fig. 2 for  $(\text{NH}_4)_2\text{SO}_4$ . The observed stretching modes have a more complicated structure. In the case of pure water (Fig. 1), they contain contributions of the symmetric and anti-



**Fig. 1:** In black (left hand scale): ATR-IR spectra of water on a germanium internal reflection element measured with  $s$  and  $p$  polarisations, respectively. In red (right hand scale): the “dichroic ratio spectrum” obtained by dividing the spectrum with  $p$  by the one with  $s$  polarisation. Also shown is a line at a dichroic ratio of 2, which corresponds to an isotropic liquid.



**Fig. 2:** In black (left hand scale): Spectra of aqueous 2M  $(\text{NH}_4)_2\text{SO}_4$  solution on a silicon internal reflection element measured with *s* and *p* polarisations, respectively. In red (right hand scale): the “dichroic ratio spectrum”. Also shown is a line at a dichroic ratio of 2, which corresponds to an isotropic liquid.

symmetric stretching modes as well as the second harmonic of the bending mode. In the case of  $\text{NH}_4^+$ -salts, the already broad peak additionally contains contributions from the three stretching modes of the tetrahedral  $\text{NH}_4^+$  ion, as well as an overtone of the bending mode [6]. For highly concentrated 2M  $(\text{NH}_4)_2\text{SO}_4$  solution, the dichroic ratio contains distinct peaks and is significantly different from the isotropic value of 2. For low ion concentrations (0.1 M  $(\text{NH}_4)_2\text{SO}_4$  and 0.1 M  $\text{NH}_4\text{NO}_3$ ),  $R \sim 2$ , i.e. the isotropic value. For intermediate concentrations (0.5M and 1M) of  $(\text{NH}_4)_2\text{SO}_4$  and 1M  $\text{NH}_4\text{NO}_3$ ,  $R \sim 1.80$  are found.  $\text{NH}_4\text{Cl}$  shows a different trend: at 1M,  $R \sim 1.9$ , but at 0.1M  $R \sim 1.7$ .

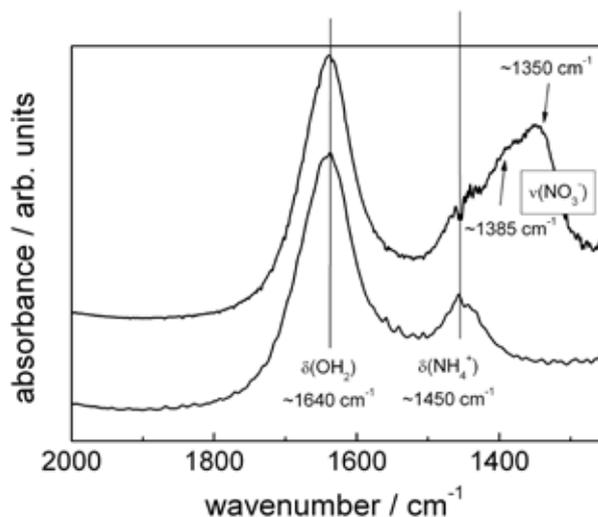
From the dichroic ratio, the orientational order parameter  $S_2 = 1/2 \cdot (3 \langle \cos^2 \theta \rangle - 1)$  can be determined, as e.g. described in [7]. Here,  $\theta$  is the angle of orientation of the transition dipole moment of the respective mode with respect to the surface.  $S_2 = 0$  corresponds to isotropic orientation. For the example from Fig. 2,  $S_2 \sim 0.05$ . For 1M  $\text{NH}_4\text{NO}_3$  and  $(\text{NH}_4)_2\text{SO}_4$  solutions of intermediate concentrations,  $S_2 \sim -0.05$  is obtained.

In order to understand the meaning of the orientational order on the molecular level, first of all, a knowledge of the orientation of the transition dipole moment in the molecular coordinates is needed. The analysis is complicated by the hydrogen bonding network, in which all of the species are involved. This H-network leads e.g. to a splitting of the degenerate stretching modes of the  $\text{NH}_4^+$  ion. The second important factor is an understanding of the averaging procedure as written in the above equation. The averaging includes all water molecules within the volume illuminated by the evanescent wave, weighted by a factor corresponding to the intensity of the evanescent wave at the position of the

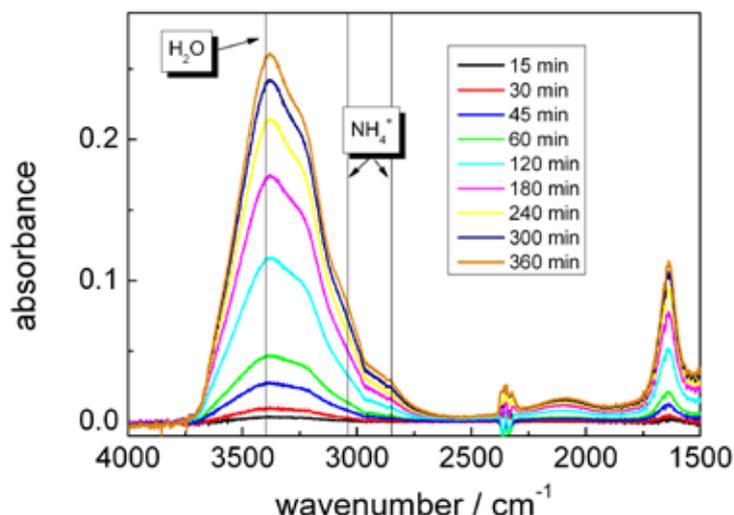
molecule. The volume included here contains  $\sim 7 \cdot 10^{18}$  water molecules. In the distance up to 0.6 nm away from the surface, we expect  $\sim 4 \cdot 10^{15}$  water molecules. Even though the effect of the molecules nearer to the surface on the spectra is larger, even a perfect order of this cannot account for the differences from isotropic behaviour as found here. Therefore, it can be clearly stated that for  $(\text{NH}_4)_2\text{SO}_4$  solutions (and 1M  $\text{NH}_4\text{NO}_3$  solution), a deviation from isotropic ordering of water on a length scale beyond the layer of adsorbed ions and bound water is observed. The order may be induced through the adsorption of ions at a solid interface. Further experiments will be needed varying the anion or cation only, also including non-absorbing ions.

In addition to the complicated NH stretching modes, the presence of the ions can be detected through other vibrational modes (Fig. 3). The  $\text{NH}_4^+$  ion shows a characteristic bending mode at a frequency  $\sim 1450 \text{ cm}^{-1}$ . Nitrate ions show a characteristic doublet peak with a maximum around  $1350 \text{ cm}^{-1}$  from the stretching modes. It should be noted that this mode is actually IR inactive and only appears if the symmetry of the  $\text{NO}_3^-$ -ion is broken e.g. due to its embedding into a hydrogen-bonded network. Because of the tetrahedral shape of the  $\text{NH}_4^+$  ion it does not show preferential orientation. However, the planar nitrate ion can show different orientations with respect to the surface. Nevertheless, the dichroic ratios observed for nitrate solutions indicate isotropic orientation of the nitrate anion.

As a second topic, initial results about the diffusion of ions under polymer films on Si substrates are reported. Here, a poly(vinyl butyral-co-vinyl alcohol-



**Fig. 3:** Examples of IR spectra of aqueous ammonium sulphate (lower spectrum) and ammonium nitrate (upper spectrum). Visible are the  $\text{OH}_2$  bending mode, the  $\text{NH}_4^+$  deformation mode and the  $\text{NO}$  stretching modes.

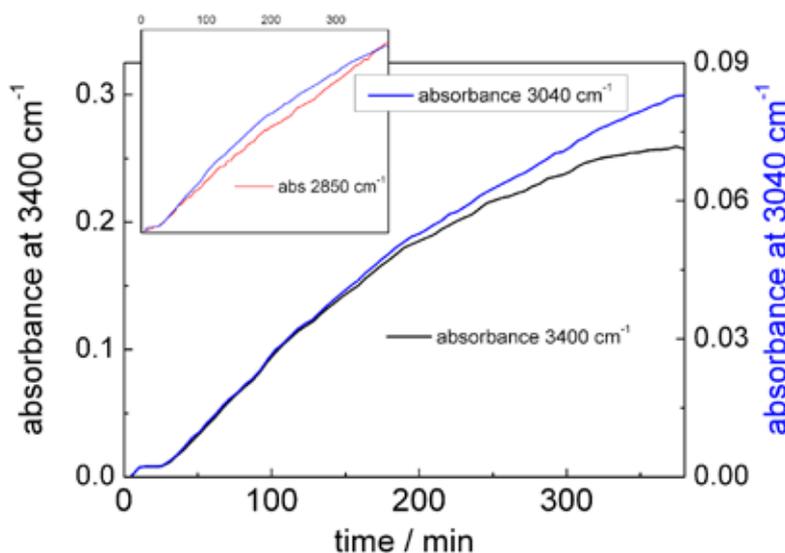


**Fig. 4:** Time evolution of the spectra after bringing into contact a 1 M  $\text{NH}_4\text{Cl}$  solution with a defect in a polymer film on a silicon crystal. The vertical lines (at 3400, 3040 and 2850  $\text{cm}^{-1}$ ) indicate the wavenumbers at which the time traces of Fig. 5 were recorded.

co-vinyl acetate) was chosen, which is also used by other groups within the department. Films of thicknesses of  $\sim 1 \mu\text{m}$  were spincoated from 2-propanol solution. After producing a defect in the polymer film, the diffusion of water and ions can be followed by IR spectroscopy as depicted in Fig. 4 and 5. Fig. 4 shows the evolution of the IR spectra as a function of time. The intensities of the modes associated with water and ions increase. In order to evaluate the diffusion of the different species under the polymer film, the absorbance at different wavenumbers has been followed with time. The resulting kinetic traces are displayed in Fig. 5 for the wavenumbers 3400  $\text{cm}^{-1}$  (associated with  $\text{H}_2\text{O}$ ), 3040 and 2850  $\text{cm}^{-1}$  (both associated mainly with  $\text{NH}_4^+$ ). After bringing the salt solutions in contact with the defect, an initial incubation phase is observed.

Later the intensity of both the bands associated with the presence of  $\text{H}_2\text{O}$  and  $\text{NH}_4^+$  start to increase. This increase however happens in a different manner for the different bands. Fig. 5 illustrates the differences: for water, the slope of the curve decreases at larger times. This decrease in the slope is more pronounced for water.

The results here clearly show that both the ions and the water penetrate the layer under the polymer film. The initial diffusion happens with the same rate both for water and for  $\text{NH}_4^+$  ions. After  $\sim 2.5$  h, the influx of water is becoming slower while the influx of ammonium ions does not significantly change. This may be related to the evaporation of water from the sample chamber, but may also have chemical reasons. A change in the hydrogen bonding structure



**Fig. 5:** Plot of the absorbance versus time at three different wavenumbers of 3400  $\text{cm}^{-1}$  (black, main graph), 3040  $\text{cm}^{-1}$  (blue, both in the main graph as well as the inset) and 2850  $\text{cm}^{-1}$  (red, inset).



would, for instance, give rise to a change in the peak broadening and consequently in a different time dependence of the different absorption modes. A better cell, more sophisticated analysis algorithms and the inclusion of the bending modes into the analysis are required in order to distinguish the different possibilities.

In conclusion, we find that in several ammonium salt solutions in contact with a silicon surface carrying its native oxide, the orientation of the water molecules is different from the isotropic orientation encountered in bulk liquids. As a second conclusion, we are able to detect ammonium ions and water diffusing simultaneously under polymer films.

In future experiments, the effect of an applied potential on both the ion diffusion and ion orientation will be studied.

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## Microscopic Understanding of the pH-dependent Structure and Stability of ZnO Surfaces in Aqueous Electrolytes

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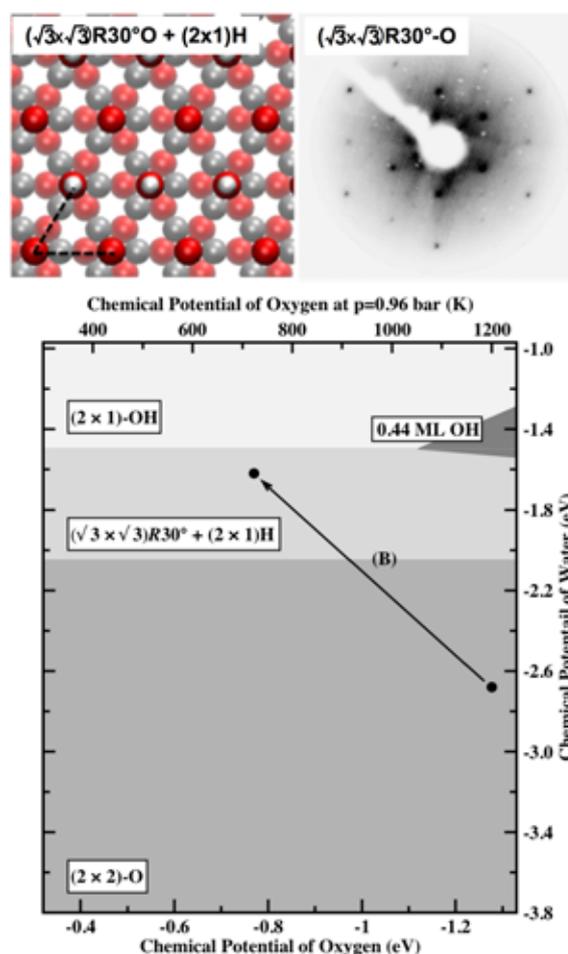
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Functional metals, which are of high industrial and technological importance, are covered by a native oxide film. The stability of these oxide films determines the performance of materials as these thin films passivate and thereby protect the surface from further oxidation. Moreover, molecular adhesion forces at interfaces between organic coatings or adhesives and engineering metals are governed by physisorption or chemisorption of macromolecules or adhesion promoting additives on these passive film surfaces [1]. In this context the stability of passivating oxides is of great interest for an effective corrosion protection [2]; an understanding of the surface chemistry on a molecular level is important for polymer adhesion on oxide covered metal substrates [3]. In particular, as zinc is a widely used metallic coating for steel, the stability of passivating ZnO films is of high technical interest. As corrosion reactions, like e.g. a cathodic delamination of organic coatings or a crevice corrosion, are accompanied by a change of the pH at the interface [1,4] it is necessary to understand the stability, surface chemistry and dissolution behaviour of ZnO as a function of the pH.

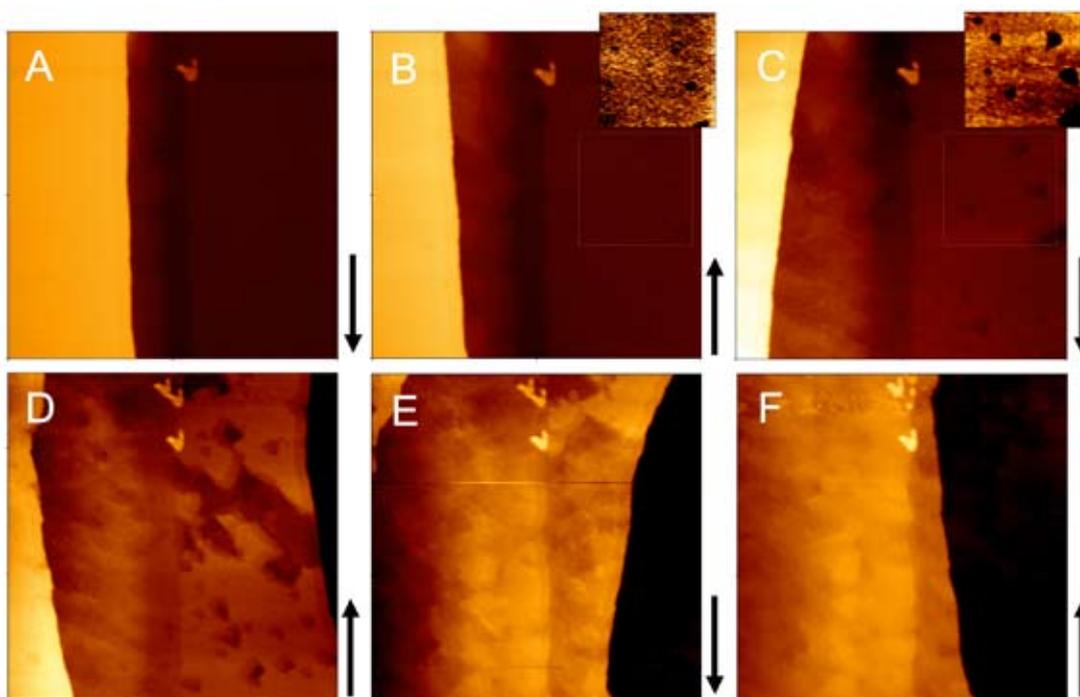
This report highlights our studies on the chemical dissolution behaviour and stabilisation mechanisms of single crystalline ZnO(0001)-Zn surfaces. Understanding the dissolution mechanism of the oxide is of high practical interest as it constitutes the main rate-determining step of corrosion reactions on zinc within the passive region, while the use of single crystals with known crystallographic orientation enables the investigation of questions fundamental to the understanding of corrosion. Furthermore, the well defined surfaces and surface structures of oxide single crystalline substrates facilitate a close interaction of experimental studies with *ab initio* simulation approaches, which is highly desirable for gaining a detailed understanding of interfacial processes on a microscopic level.

Single crystal ZnO(0001)-Zn surfaces were prepared by annealing in humidified oxygen atmosphere [5]. The resulting model surfaces are single crystalline, feature very large atomically flat plateaux in the  $\mu\text{m}$  range and show the super structure of a stabilising ad-layer in LEED. The step heights are typically between 4 to 10 nm. All relevant experimental details can be found in the literature [6]. One of the intriguing questions was to identify and understand the structure and morphology of the stabilising ad-

layer structure on the surfaces prepared with our ambient atmosphere approach. For this purpose we investigated the morphology of the polar ZnO(0001) surface in a humid oxygen environment by combining our experimental findings with *ab initio* theoretical investigations. LEED results suggest the presence of a  $(\sqrt{3}\times\sqrt{3})R30^\circ$  superstructure on top of the Zn-



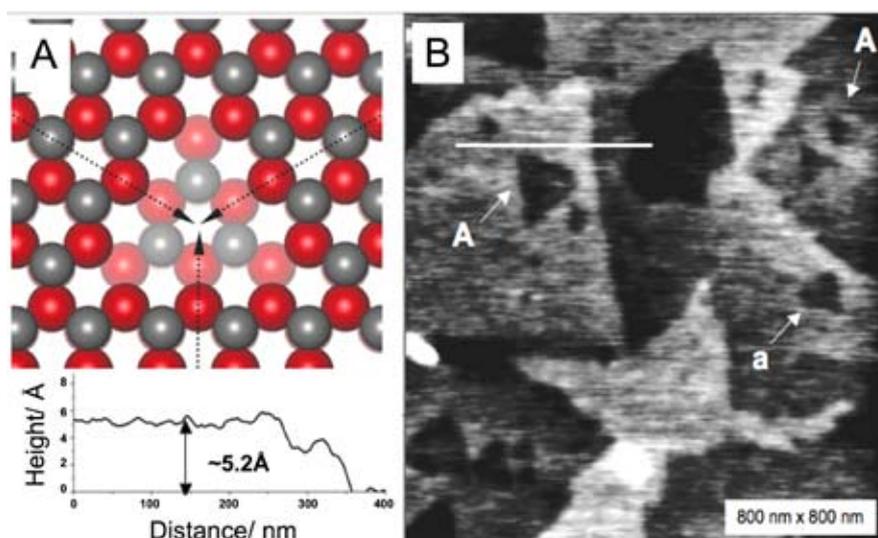
**Fig. 1:** (Top) On the left hand side a top view of the structure found to be stable on the ZnO(0001) surface for oxygen rich conditions at  $p = 0.96$  bar oxygen partial pressure and 4 mbar water pressure is depicted. Red circles denote O atoms, grey circles denote Zn atoms, and smaller white circles denote H atoms. On the right hand side, LEED images obtained at 63 eV showing the  $(\sqrt{3}\times\sqrt{3})R30^\circ$  periodicity for the surface prepared in humid oxygen atmosphere are depicted. (Bottom) Surface phase diagram of stable structures (gray shaded areas) of ZnO(0001)-Zn in equilibrium with a humid environment. The chemical potential of oxygen is expressed also in terms of temperature at the used experimental oxygen partial pressure of  $p = 0.96$  mbar. Experimental observations are marked by black dots and arrows.



**Fig. 2:** This figure represents the evolution of the ZnO(0001)-Zn surface topography due to switching the pH from 4.0 to 3.6. The step height is 4.8 nm. The insoluble impurities are a good guide for the eyes. Scan size for topography images (A-C) was  $1\mu\text{m} \times 1\mu\text{m}$ . As the dissolution front was running out of the image, the scan size was enlarged to  $1.2\mu\text{m} \times 1.2\mu\text{m}$  for (D-E). The arrows indicate the scan direction. (A) Represents the scan directly after changing the pH of the base solution from 4.0 to 3.6, which corresponded to a pH of 3.9 in the cell. The dissolution started to proceed immediately with a higher speed as can be seen in the next scan (B) that was finished 3 minutes later. The small inlet clearly shows, that first pits are forming on the plateaux. The next scans (C-F) show the evolution (+3 minutes per scan) of the surface structure at the final pH of 3.6. The inlet in (C) shows that the initial pits are growing in x-y direction.

terminated surface. Aiming to identify its geometry, we performed *ab initio* calculations considering possible geometries with surface unit cells consistent with the observed LEED pattern. Combining these results with atomistic thermodynamics considerations allows us to bridge the temperature and pressure gap between theory (ultrahigh vacuum,  $T = 0\text{K}$ ) and experiment. A direct comparison between the theoretical and experimental observations is realised by considering for the surrounding gas atmosphere chemical potentials, which concur with the experimentally used environment. Using this combined experimental/theoretical methodology we were able to identify a phase featuring alternating rows of oxygen atoms and hydroxyl groups as being thermodynamically favoured for the conditions of the experimentally used gas atmosphere (4% water vapour at a final temperature of  $450^\circ\text{C}$ ) within the *ab initio* derived phase diagram depicted in Fig. 1. This phase contains  $1/6\text{ML OH}$  and  $1/6\text{ML O}$ . It consists of a  $(\sqrt{3} \times \sqrt{3})R30^\circ$  backbone of O atoms, with H atoms adsorbed on top of them in a  $(2 \times 1)$  arrangement, in good agreement with our experimental LEED data. The competition between the  $(2 \times 2)\text{-O}$  phase, which is also included in the phase diagram, and triangular reconstructions discussed in the literature [7] is still a subject of investigation and will be published elsewhere [8].

Studying the dissolution mechanism on these model surfaces promoted several fascinating results. Within a pH range of 11 to 5.5 significant changes of the topography could not be observed. Below this pH level, within a pH range from 5.5 to 3.8, the dissolution started to proceed exclusively along the pre-existing edges but not on the terraces. In Fig. 2 A the topography of an etched ZnO(0001) surface at pH 3.9 is depicted, where it can be clearly seen, that dissolution on the terraces is not observable. Up to this point the dissolution was exclusively running along the pre-existing step edges. Considering the crystallographic structure of the ZnO it is quite clear why the etching process preferably proceeds at the edges of the structure. Along these step-edges the single crystal features low-index  $11\bar{2}0$  or  $10\bar{1}0$  surfaces, in which the oxygen as well as the zinc atoms have one dangling bond. For this reason it can be assumed, that these low-index surfaces are thermodynamically less stable compared to the stabilized polar surface. Furthermore, adsorption of protons at the polar ZnO(0001) surface is not favourable as the surface is positively charged within acidic electrolyte. For even lower pH values in the range of  $\sim 3.8\text{-}3.6$  the dissolution started to proceed also on the terraces as can be clearly seen in Fig. 2(B-F). When the pH was further decreased the attacks on the terraces significantly increased the nano-roughness on the terraces.



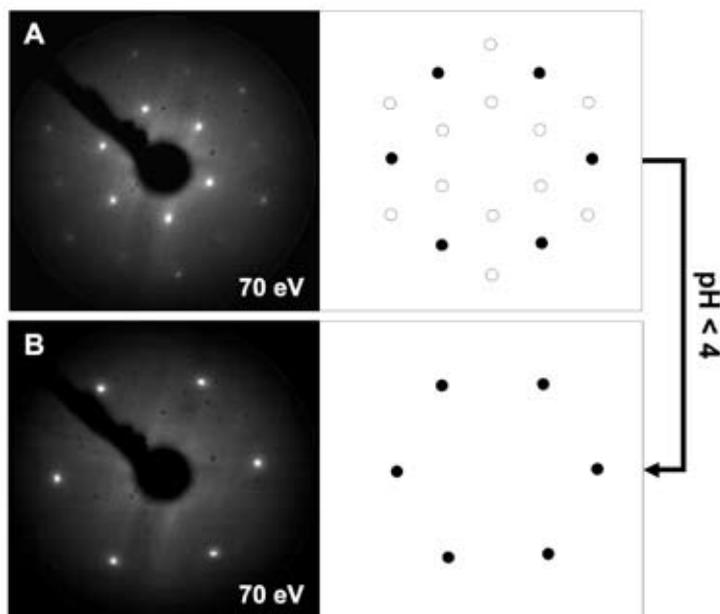
**Fig. 3:** (A) shows an equilateral triangle, which is delimited by symmetry equivalent step orientations. Only triangular features allow a termination by the same step orientation at all three sides. (B) Ex-situ AFM topography scan of a surface etched below pH 3.8. Triangular features on the surface are clearly visible and crystallinity is evident. As can be seen clearly, triangular features on “A”-terraces are rotated 60° with respect to triangles on “a”-terraces. The line-profile (measured as indicated) shows that the steps-heights correspond with the expected Zn-O double layer height.

Interestingly, it was observed that the surfaces show characteristic crystalline features even at pH-values as low as 3. These features turned out to be very different compared to the atomically flat structure at higher pH values. The plateaus show various steps resulting in a high density of edges. Furthermore, equilateral triangles reflecting the hexagonal surface structure were consistently found on these surfaces. The measured step heights of  $2.6 \pm 0.2 \text{ \AA}$  and  $5.2 \pm 0.2 \text{ \AA}$  are consistent with the expected values for the ZnO crystal structure (see Fig. 3B). In fact, the triangular features and the high density of steps are comparable with the surface structure found for UHV prepared surfaces. DFT calculations of G. Kresse et al. [9] revealed that the formation of triangular reconstructions enables an energetic stabilization of the ZnO(0001)-Zn surface in the absence of a sufficient hydroxide coverage. The energy gain due to the formation of triangular features could be clearly related to a decrease of the Madelung energy. Formation of triangular structures reduces the number of repulsive next nearest neighbour interactions in the second coordination shell. In electrolyte solutions the formation of triangular reorganizations is possibly also favourable due to a gain of Madelung energy at these very low pH values. However, in aqueous electrolyte a gain of Madelung energy is difficult to be quantified because the adsorption of oriented water dipoles could lead to a much lower energy gain related to the Madelung Energy. Therefore it is reasonable to consider possible other reasons for the stability of triangular features as well.

In electrolyte solutions triangular features are moreover promoted based on the delimitation by lowest energy step edge configurations. The

triangular geometry of the nano-terraces supports this hypothesis. If one specific step orientation is most stable - and thus most favourable - it will exist on the closed packed surface within the hexagonal wurtzite structure exactly along three symmetry equivalent directions that are rotated 120° with respect to each other (see Fig. 3A). Thus the formation of equilateral triangular features itself indicate one specific step orientation. This conclusion can further be experimentally supported if the triangular features are rotated by 60 degrees with respect to each other on plateaux that are separated by one double layer of ZnO. The same step termination along the next Zn-terminated layer must be rotated by 60° because ZnO features an [-A-B-a-b-A-B-a-b-] structure of hexagonally closed packed planes of Zn (A/a) and O (B/b) along the c-axis. Zinc planes that are separated by one ZnO double layer are rotated by 60 degrees with respect to each other. Thus, an “A” Zn-plane is rotated by 60° with respect to an “a” Zn-plane and consequently equivalent edge terminations must be rotated by 60 with respect to each other as well. Exactly these surface structures could be observed for all ZnO(0001)-Zn crystal that were immersed into electrolyte solutions with pH values lower than 3.8 as can be seen in Fig. 3B. Therefore it can be concluded that the triangles are delimited by one specific symmetry-equivalent and most probably closed packed surface orientation. Moreover, triangular structures provide a kinetic stabilization because these triangles do not feature any kink-sites as can be seen in Fig. 3.

Complementary to the AFM studies, ex-situ LEED investigations were performed, which confirmed the presented AFM-investigation. Below pH 4 a



**Fig. 4:** (A) Characteristic LEED pattern of the ZnO(0001)-Zn surfaces after immersing into solutions of the different pH values from 11 to 4. (B) After immersing the surface into solution with  $pH < 4.0$  the LEED characteristics of the surface changed significantly. It can be seen clearly that specific pattern at 70eV disappeared as shown in the schematics. This clearly indicates that the  $(\sqrt{3} \times \sqrt{3})R30^\circ$  superstructure is irreversibly destroyed due to immersion of the surface at pH values lower than 4.0.

significant change in the surface crystallography could be detected, which is indicating the dissolution of stabilizing hydroxid/ oxygen adlayer at the polar surface (see Fig. 4). Based on these findings it can be concluded that the surface structure on the plateaux is not sufficiently stabilized at these acidic pH-values. The obviously very stable adlayer is dissolved at these low pH values due to protonation of the surface hydroxides. The surface energy will be quite high due to these unfavourable conditions. Hence an energetically more stable situation will be promoted and energetically favourable triangular structures form and sufficiently stabilize the surface in acidic environment. Hence, the ZnO(0001)-Zn surface changes its stabilization mechanism below this pH value due to first the minimization of the Madelung energy and second the preferable formation of stable  $(10\bar{1}0)$  step orientations by the evolution of

triangular reorganizations. In fact this is a very interesting result, because this is the first in-situ observation of a change of the stabilization mechanism of a polar oxide surface.

To summarize, these results promote a microscopic understanding of the pH-dependent structure and stability of zinc-oxide surfaces in aqueous electrolytes. Moreover, these results provide the basis for a synergistic bridging of novel ab-initio based multi-scale modelling methods and experiment even within electrolyte environment. Only a combined approach of theory and experiment, as provided by the cooperation between the CM and the GO department, allowed for an atomic-scale understanding of the surface morphology. A detailed discussion of the results can be found in the literature [6].

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## Towards Corrosion Control from First Principles

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Corrosion is a detrimental process, caused by reactions with the environment. When a metal comes into contact with an oxidising environment, such as e.g. water, it (the metal) does not correspond any longer to a thermodynamically stable phase. A corrosion cell builds-up (Fig.1a), in which the liquid acts as an ion-conductor, while the metal is responsible for transport of electrons from a local anode, at which metal ions are oxidised and go into solution, to a local cathode, at which ions are reduced and electrons consumed. As long as the flux of metal ions going into solution exceeds the flux of metal ions being reduced at the surface, the material corrodes.

A material can be protected against corrosion, as achieved e.g. by galvanisation of steel. Here the applied zinc layer (Fig.1b) offers threefold protection: (i) it constitutes a protective layer, which shields the steel surface from the environment, (ii) due to the lower corrosion potential of zinc the zinc layer is the first to corrode following a breakthrough, (iii) the Zn-corrosion products precipitating on the steel surface inhibit oxygen reduction significantly, thus relieving the corrosion pressure from the zinc coating.

Improving corrosion protection is, however, often a question of trial and error approaches, largely based on empirical observation rather than sound theoretical understanding. The identification and accurate characterisation of elementary processes taking place at the solid/liquid interface would constitute an essential step towards gaining in-depth insight into the functionality of a corrosion cell considering, that the plethora of possible elementary

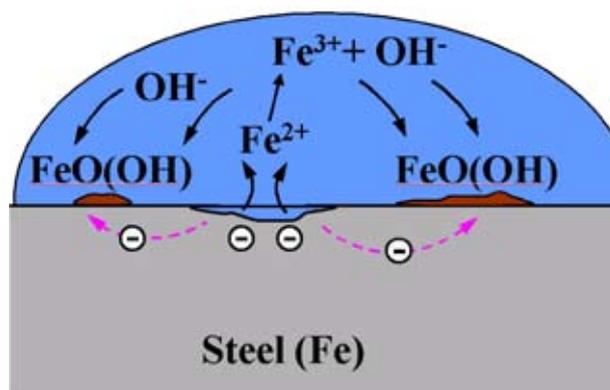
processes at the interface determines the ease with which a metal atom leaves the solid surface to go into solution and their interplay accounts, in turn, for the corrosion rate.

**Methodological aspects.** A meaningful description of the elementary processes occurring at the solid/liquid interface requires the accuracy of a quantum mechanical description. Density-functional theory (DFT) is the electronic structure method of choice, having been applied often and successfully to the description of surface and interface phenomena. The problem at hand remains, however, a very challenging undertaking, mainly due to the prominent role which water plays in a corroding system and the required use of large unit cells.

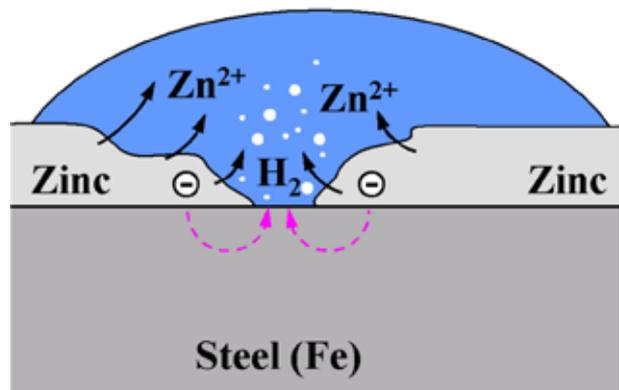
A first step towards tackling the problem and making the computations feasible was the development of a methodology which allows us to access parameters relevant to corrosion, such as the electrode potential of the solid surface or the pH-value of the liquid phase, from first-principles. Our approach is based on concepts originally developed for the defect chemistry of semiconductors and is safely based on the formation enthalpy of ions in a host matrix (here: solution). Such formation enthalpies are accessible to DFT calculations [1]

$$E^f [X^q, (\mu_i, E_F)] = E_{\text{tot}}[X^q] - E_{\text{tot}}[\text{bulk}] - \sum_i n_i \mu_i + qE_F$$

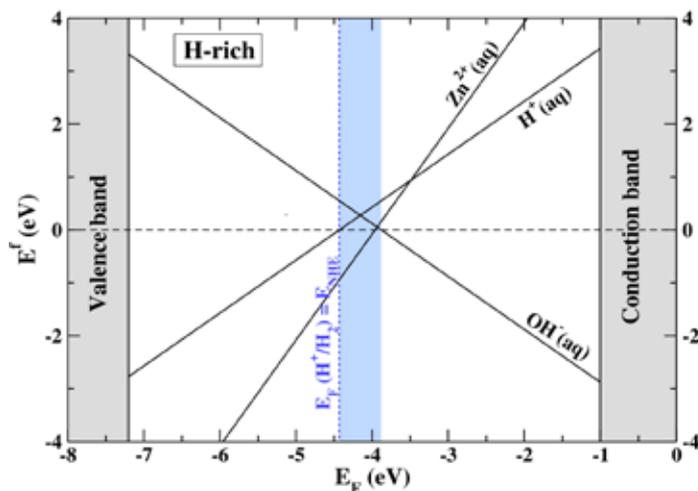
They require the consideration of a unit cell containing a defect/ion with charge  $q$ ,  $E_{\text{tot}}[X^q]$ , of a defect free unit cell,  $E_{\text{tot}}[\text{bulk}]$ , and depend on the chemical potentials of the involved species,  $\mu_i$ , as



**Fig. 1a:** Schematic picture of a steel corrosion cell: Metal ions go into solution at the local anode and are reduced at the local cathode to form corrosion products.



**Fig. 1b:** Schematic picture illustrating the role of zinc in steel corrosion protection: Due to its lower corrosion potential zinc offers cathodic protection to steel, even upon a breakthrough of the zinc protective layer.



**Fig. 2:** Formation energies of ions as a function of the Fermi energy within the band gap of water. The region of water stability is shaded light blue. The Fermi Energy corresponding to the Normal Hydrogen Electrode is denoted by the dashed dark blue line.

well as on the chemical potential of the electrons,  $E_F$ . In any given system, the defect with the lowest formation energy is identified as the one being thermodynamically stable for the given conditions, i.e. chemical potential and Fermi energy. Knowledge of the formation enthalpy also allows the determination of the concentration [1] the given defect can achieve in the system under consideration, since this concentration is proportional to a Boltzmann factor.

**Objective.** To demonstrate the potential and strength of our novel approach we initially utilise tabulated solvation data [2] to calculate the formation energies for ions in water and subsequently use them

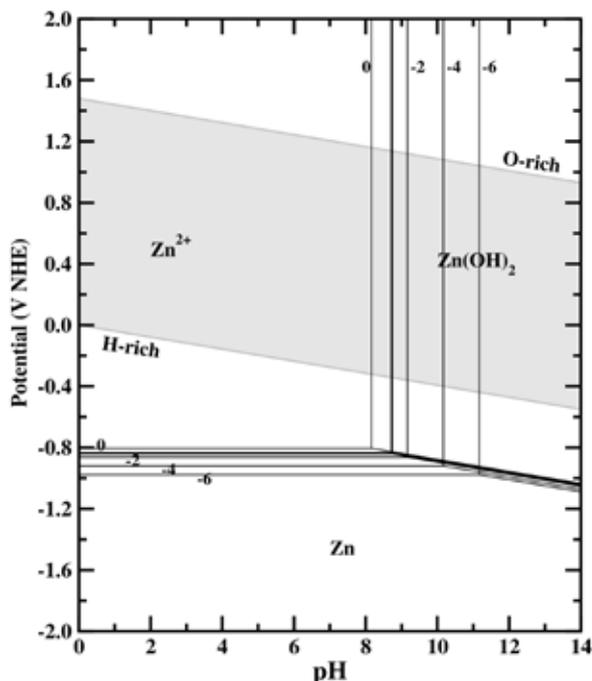
to construct Pourbaix diagrams, which are a well established tool in corrosion science, for a wide range of metals.

The key quantity which enters our new approach is the hydration energy of ions in the water host matrix. This necessarily means that an accurate and reliable description of the water matrix is crucial and needs to be carefully tested. In a first approximation we therefore model the water host as a form of ice, where the water molecules are arranged on a regular lattice. We investigate the accuracy which can be achieved by employing present day DFT-functional by comparing calculated and measured elastic, thermal, vibrational and dielectric properties of ice.

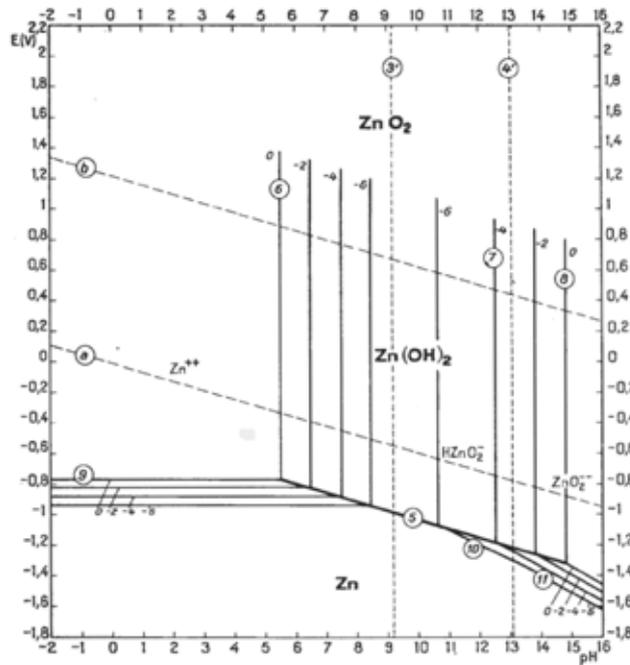
**The Pourbaix diagram of zinc.** Pourbaix diagrams are an important tool to identify the conditions under which a surface is thermodynamically stable against corrosion.

So far such diagrams are constructed based on experimental data which are difficult to calculate by first-principles. In the following we demonstrate the reliability of our approach by constructing a Pourbaix diagram for Zn (Fig. 3).

We first evaluate the stability of  $H^+(aq)$  and  $OH^-(aq)$  within the band-gap of water, by investigating their respective formation energies for a given chemical potential (e.g. hydrogen-rich conditions) as a function of the Fermi Energy, which is viewed as a variable within the band-gap (Fig. 2). We identify the conditions for which water is a stable phase (blue marked narrow region within the band-gap). Keeping



**Fig. 3a:** Pourbaix diagram for Zn obtained by the alternative approach from the formation energies of ions.



**Fig. 3b:** Pourbaix diagram for Zinc; taken from Ref. [3].



in mind that the  $pH$ -value is defined as the negative logarithm of the  $H^+(aq)$  ion concentration, we are able to link the  $pH$ -scale to the formation energy of the  $H^+(aq)$  ion, allowing the determination of  $pH$ -values directly from DFT calculations. On the other hand, knowing that the normal hydrogen electrode ( $E_{NHE} = 0\text{ V}$ ) is defined for H-rich conditions and  $pH = 0$ , allows us to identify the value of the Fermi Energy marked by a dashed blue line in Fig. 2 as  $E_{NHE}$  and use it as a reference when determining an electrode potential, in analogy to experimental practice.

The Pourbaix diagram for Zinc, which we construct using our new approach is shown in Fig. 3a and found to be in very good agreement with experiment (Fig. 3b) [3]. The differences observed on the  $pH$ -scale are understood in terms of the water's stability region within the band-gap, which comes out to be slightly too small for the formation energies used here. A similar agreement has been also found for an extensive set of other metals (e.g. Fe, Cu, Mg).

**Bulk properties of ice.** The central role which water plays with respect to the hydration energies of ions, necessitates a careful investigation of the accuracy and reliability of the description we can achieve for the water matrix using present day DFT-functionals. We start out by modelling the water matrix as a form of ice, which will allow us to compare calculated bulk properties to experimental findings.

We focus our investigations on hexagonal ice, which is the form natural ice is found to have. However, hexagonal ice is known to possess two phases, with the phase transition between them occurring at  $\sim 72\text{ K}$ . The oxygen atoms in both cases are arranged on a wurtzite lattice and the difference between the phases arises from the behaviour of the corresponding protons. The high temperature phase,  $lh$ , exhibits proton disorder, meaning that the protons are able to interchange their position and are thus smeared out over their lattice sites. In contrast, the protons in the low temperature phase of hexagonal ice, ice XI, are frozen in at their lattice positions.

The DFT calculations are performed on the exchange-correlation functional level of a generalised gradient approximation (GGA-PBE), as implemented in the SFHInGX program library. We use norm-conserving pseudo potentials and super-cells containing from 8 up to 32 water molecules, arranged on a regular lattice. We determine the equilibrium lattice parameters to be  $a = 4.41\text{ \AA}$ ,  $b = \sqrt{3} \cdot a$  and  $c/a = 1.631$ , in accord with other theoretical investigations and in reasonable agreement with experiment. We also determine the theoretical bulk modulus by employing the Murnaghan equation of state to be  $B = 14.4\text{ GPa}$ , which differs significantly from the experimentally reported bulk modulus of  $8.9\text{ GP}$  ( $T = 257\text{ K}$ ). We find similar discrepancies when we compare the calculated and measured elastic

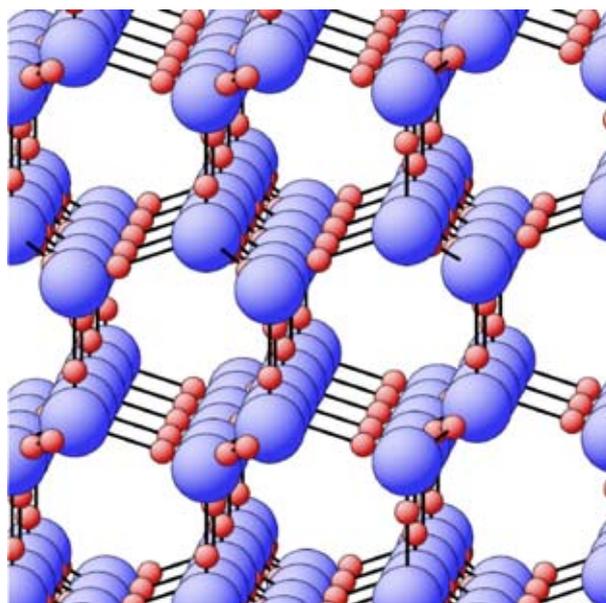


Fig. 4: Structure of hexagonal ice. O: blue, H: red.

constants, i.e. both the bulk modulus and the elastic constants we obtain are bigger than the known experimental values.

These findings cast some doubt on the accuracy of GGA-DFT to describe the elastic properties of ice, since they lead to the conclusion that DFT-ice is much too hard. However, in DFT calculations we describe the low temperature phase of hexagonal ice (ice XI) in which the protons are frozen in at their lattice positions. Unfortunately, this proton ordered low temperature ice phase it is very difficult to prepare experimentally [4], requiring e.g. the use of catalysts. The predominant part of experiments are therefore performed using proton disordered ice samples (i.e. the high temperature phase,  $lh$ ).

To investigate the question if the observed discrepancies are indeed due to the GGA-functional or a consequence of neglecting relevant free-energy contributions we investigate the effect thermal (i.e. vibronic) excitations have on this result. In a first step we therefore study the thermal expansion of the lattice, i.e. the change of lattice dimensions with temperature. Within the quasi-harmonic approximation we calculate *ab initio* phonon spectra for several different volumes  $V$ . These lattice excitation energies enter the partition function and provide a temperature dependent expression for the vibrational entropy [5]. By adding this contribution to the  $T=0\text{ K}$  DFT result we obtain the Helmholtz free energy  $F(T, V)$ . The thermal expansion can be now directly obtained by determination of the volume  $V$  which minimises  $F(T, V)$  for a given temperature  $T$ .

The resulting *ab initio* derived thermal expansion of the lattice and the linear expansion coefficient both agree quantitatively and qualitatively with

measurements for ice. Despite our calculated ice still being too hard, this observation gives confidence, that DFT-GGA is correctly describing the main features of ice. As will be shown in the following, a further improvement is achieved, when a more sophisticated description of the vibronic excitations is used.

Up to this point we neglected to consider the contribution of anharmonic effects to the vibrational entropy. We now also include anharmonic contributions by employing the method of thermodynamic integration [6]. Within this approach molecular dynamic simulations are performed on a potential

$$U(\lambda) = \lambda U^{\text{full}} + (1-\lambda)U^{\text{harm}},$$

approaching the full potential adiabatically. Integration over different  $\lambda$  values allows then the determination of the Helmholtz free energy. The consideration of these anharmonic vibrational contributions turns out to be essential and reduces the hardness of ice. Use of this advance methodology shifts the bulk modulus towards the experimental value, rendering it  $B = 10.3$  GPa at  $T = 200$  K. A further important aspect in correctly predicting solvation energy is an accurate description of the electrostatic screening of charged ions in water. First calculations performed using unrelaxed geometries indicate that the screening properties of ice are also reproduced quite well. We therefore conclude that the description of ice on the GGA-PBE level is accurate enough, which allows us to now turn our attention towards obtaining the hydration energies of charged ions from our *ab initio* calculations.

**Discussion and conclusions.** We have developed an approach which enables us to study questions from the realm of corrosion on the level of *ab initio* calculations. Being similar to the methodology used in the defect chemistry of semiconductors the new procedure is safely based on the formation/solvation energy of ions in the host matrix/solvent. The electrode potential and the pH-scale can be easily understood and linked to DFT-calculations within the new method. The reliability of our approach was demonstrated by the example of a Pourbaix diagram for zinc, constructed using the hydration energies of the  $\text{H}^+(\text{aq})$ ,  $\text{OH}^-(\text{aq})$  and  $\text{Zn}^{2+}(\text{aq})$  ions and which

compares remarkably good to the experimentally derived diagram.

The formation energy of ions in water being the key quantity entering the new approach requires an accurate and reliable description of the water matrix, a point which had to be carefully tested. To this end we investigated the accuracy that we can achieve in the description of the elastic, thermal, vibrational and dielectric properties of hexagonal ice using GGA-PBE. We were able to explain the discrepancies we observed initially between our calculated and the measured ice bulk properties, as arising from the comparison of the low temperature proton ordered phase of hexagonal ice (ice XI), which was considered in the calculations and the high temperature proton disordered phase of hexagonal ice (ice Ih), which was considered in experiment. Anharmonic contributions were shown to be large and particularly important to achieve an accurate description of quantities like the bulk modulus or the elastic constants of the high temperature hexagonal ice phase.

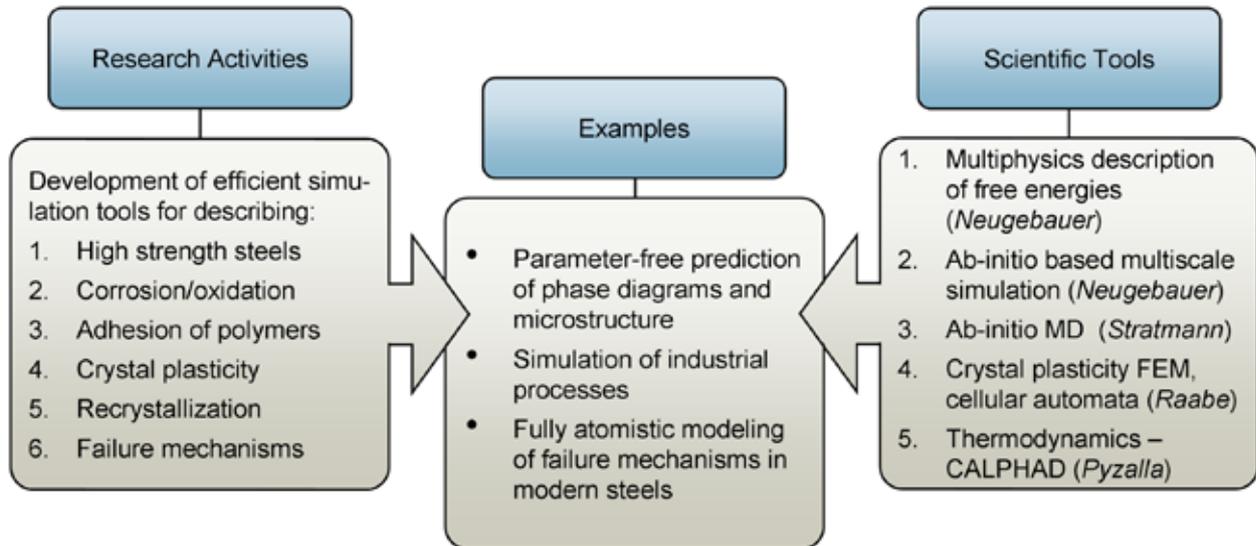
These investigations of ice give us the confidence that the description we are able to attain for a water containing system by GGA-PBE calculations is sufficiently accurate and will be an excellent starting point to tackle the wide variety of corrosion science problems.

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# Scale-Bridging Simulations of Materials



## Selected Highlights 2007/2008

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## *Ab initio* Determination of Phase Transitions with High Accuracy: The Complete Set of Free Energy Contributions

T. Hickel, B. Grabowski, F. Körmann, M. Uijtewaal, J. Neugebauer

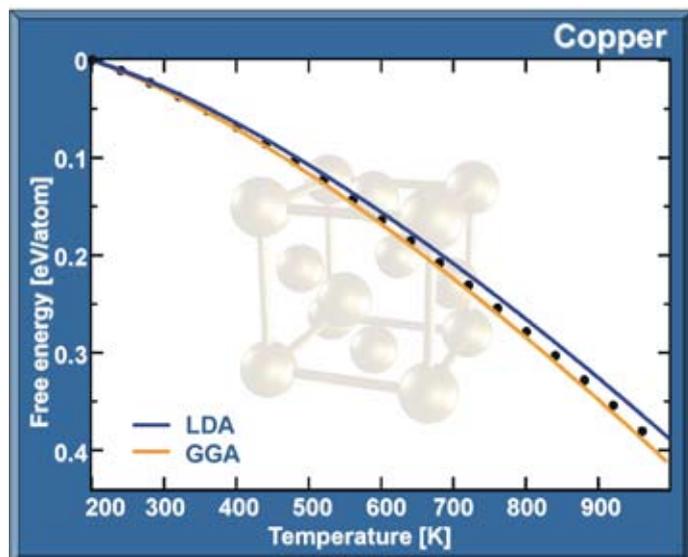
Department of Computational Materials Design

Mechanical properties of metals are closely related to the complex phase diagrams of these materials. Already elementary iron can be observed in four different solid phases. The stiffness of steels is related to the occurrence of further phases due to the inclusion of carbon and the formation of carbides. In particular, in high-strength steels the mechanical properties are influenced by the formation of a martensite, which is derived from the austenite by a diffusionless (martensitic) transformation. For a materials design it is, therefore, decisive to obtain thermodynamic information on the stability of phases and transitions between them. For this purpose, approaches such as CALPHAD [1] became important, which are based on calorimetric data and sophisticated inter- and extrapolation schemes of free energies. Despite its success a further development of the method with respect to predictive power (e.g. for new alloys/phases), efficiency (e.g. by reducing the experimental input), applicability (e.g. for metastable precipitate phases) and extension to new thermodynamic quantities (e.g. pressure dependence) is crucial.

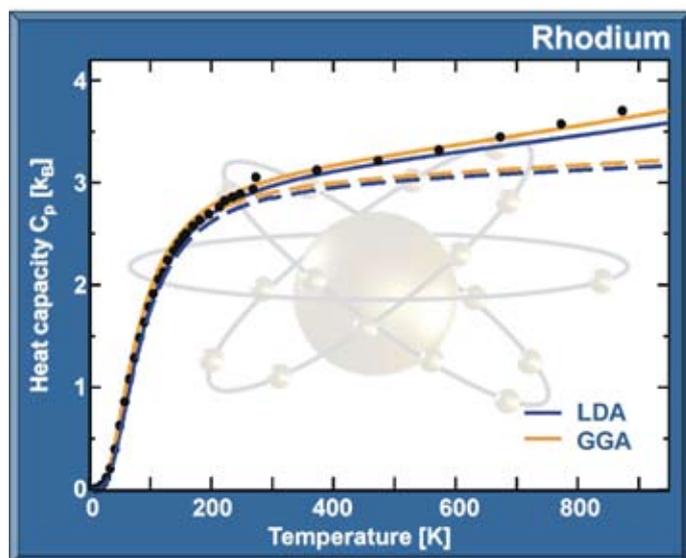
A promising approach is a partial or complete replacement of the experimental and empirical input to CALPHAD by *ab initio* data. For this purpose a modelling of the complete temperature dependence of free energies within density functional theory (DFT) is desired. The conceptual difficulty of such an *ab initio* approach arises from the fact that DFT is traditionally only used to calculate ground-state properties. For the derivation of free energies, on the other hand, one is confronted with a large variety of thermodynamic excitations (e.g., vibronic, electronic, magnetic excitations, chemical disorder, point defects and interactions between them), which in principle should all be taken into account. A restriction to the most relevant excitation processes for the problem at hand is often necessary, since the additional computational effort is severe and in some cases inhibited an *ab initio* derivation in the past. Nevertheless, we will show in the following that energy differences of a few meV can be decisive for proper predictions of materials properties and that in order to achieve these energies, the consideration of several processes is required and needs to be performed with high demands on the accuracy.

It is well known that for many material systems the excitation of **lattice vibrations** (phonons) yields the most dominant contribution to the entropy. These vibrations give rise to lattice expansions and substantially contribute to enthalpies, heat capacities and several other thermodynamic properties. *Ab initio* phonon frequencies can be obtained employing the quasiharmonic approximation [3]. Here, one makes use of the fact that the forces caused by the displacement of atoms are also accessible in DFT. The eigenvalues of the such derived dynamical matrix determine the vibrational contribution to the free energy  $F(T, V)$ . It is important to note that the quasiharmonic approximation already includes certain **anharmonicities**, if the dynamical matrix is determined for the volumes  $V(T)$  given by the thermal expansion, which in turn is self-consistently derived from  $F(T, V)$ . Nevertheless, a harmonic potential for individual atoms is still assumed and it was completely unclear if the required accuracy can be achieved with such an approximation.

We have therefore compared the temperature dependence of the derived free energy with the results of the CALPHAD method for a representative set of non-magnetic (no magnon-related entropy contributions) pure metals (no configurational entropy contributions). The study included metals such as copper (see Fig. 1), lead, aluminum, rhodium, silver,



**Fig. 1:** *Ab initio* free energy of copper, as obtained within the quasiharmonic approximation. The DFT results in LDA (blue line) and GGA (orange line) are compared to CALPHAD data (dots).



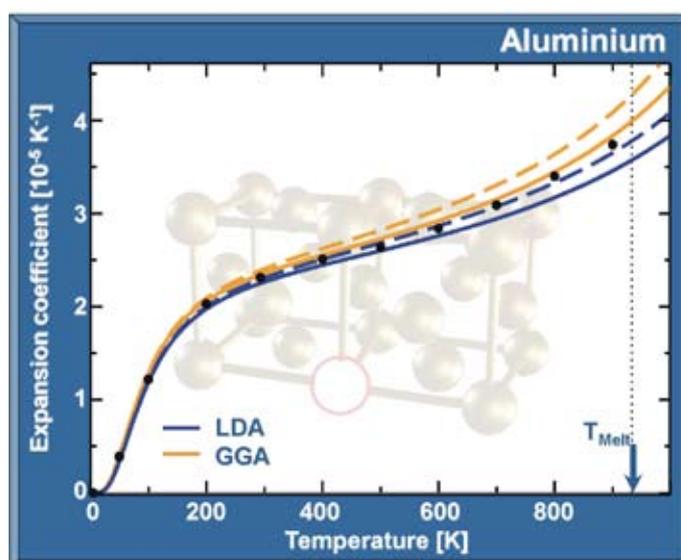
**Fig. 2:** *Ab initio* heat capacity of rhodium solely based on the quasiharmonic approximation (dashed lines) and with electronic excitations additionally taken into account (solid lines). The DFT results in LDA (blue line) and GGA (orange line) are compared to experimental data (dots).

gold and others. We systematically observed a very good agreement of the *ab initio* free energies with the CALPHAD data [4]: Over a temperature range of more than 1000 K the deviations were less than 5% ( $\approx 20$  meV/atom). In extensive convergence tests (up to  $2 \times 10^5$  k-points,  $10^6$  grid-points for augmentation charges and super-cells of 500 atoms), we have ensured that our numerical error bar is with  $< 1$  meV/atom considerably lower. As a consequence, the only two explanations for remaining discrepancies between theory and experiment are: (i) the exchange-correlation (XC) functional, for which an analytical approximation cannot be avoided in DFT, or (ii) the neglect of physical effects by the quasiharmonic approximation.

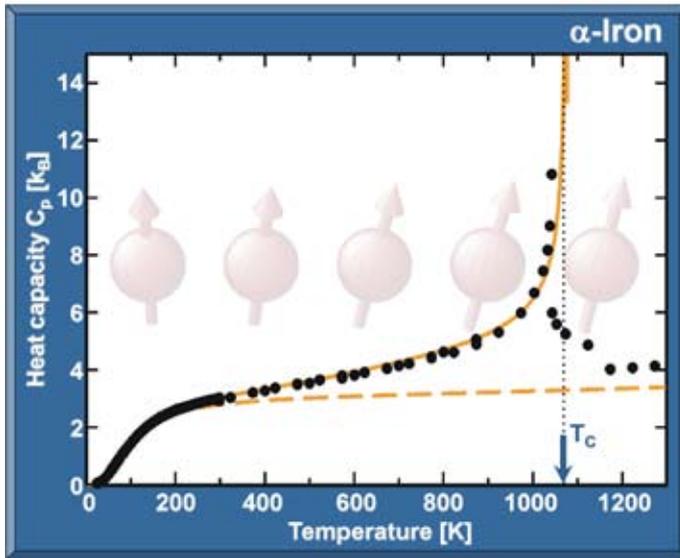
In the case of copper (Fig. 1) the free energies are systematically over-/underestimated by LDA/GGA (the most commonly employed exchange-correlation functionals). Since a physical effect can hardly be responsible for this opposite behaviour, we conclude that the discrepancies are predominantly determined by the choice of the XC-functional. Our in-depth analysis of the deviations has indeed shown that the errors are largely due to the limited accuracy of the  $T = 0$  K potential energy surface  $E(V)$  obtained from LDA and GGA. A similar relation between the experimental, the LDA and the GGA data has been observed for a large variety of materials, temperatures and thermodynamic quantities. Hence, these investigations indicate that the difference between the two XC-functionals can serve as an *ab initio* error bar.

Rhodium is a typical example of a metal, for which the quasiharmonic lattice vibrations turned out to be not the only relevant excitation process. In this case the deviation of LDA and GGA from the CALPHAD free energy is in the order of 3%. However, in the second derivatives of the free energy the effects become much more apparent: One observes for rhodium that the quasiharmonic approximation (dashed lines in Fig. 2) significantly underestimates the experiment, regardless which XC-functional is used. Since rhodium has a high electronic density of states close to the Fermi level, **electronic excitations** were a probable candidate for a missing physical effect. Indeed, after including these processes into our DFT calculations [2] the results for the heat capacity of rhodium substantially improved (solid lines in Fig. 2). Systematic studies showed that the combined consideration of phonon and electronic entropy contributions yields excellent free energies also for other transition metals [4].

We have further had a closer look at the thermodynamic properties of aluminium, which is often used as a testbed for thermodynamic modelling, due to its simple electronic structure and its high purity [5]. In this case the quasiharmonic approximation yields free energies that hardly differ between LDA and GGA. The discrepancies, again, become much more apparent in a derived quantity such as the expansion coefficient. We figured out that their qualitative behaviour close to the melting point ( $T_M = 934$  K) can only be reproduced, if **non-linear**



**Fig. 3:** *Ab initio* lattice expansion of aluminium based on the quasiharmonic approximation (dashed lines) and with vacancies & explicit anharmonicities additionally taken into account (solid lines). The DFT results in LDA (blue line) and GGA (orange line) are compared to experimental data (dots).



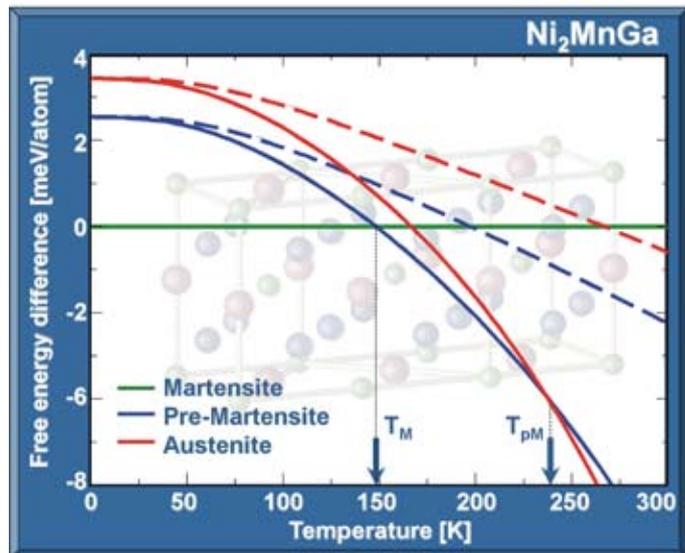
**Fig. 4:** *Ab initio* heat capacity of bcc iron based on the quasiharmonic approximation (dashed line) and with magnetic excitations additionally taken into account (solid line). The DFT-GGA results are compared to experimental data (dots).

(i.e. volume-dependent) **Grüneisen parameters** are taken into account (dashed lines in Fig. 3). While this significantly improved the agreement to the experiment, quantitative discrepancies, in particular of the GGA, remain. The relevance of additional excitation processes close to the melting point is already intensively discussed since the 1960s, when a non-Debye behaviour was recognized for aluminum. Even today this issue is not settled and there is an open controversy on the effect vacancies and anharmonic vibrations [6] have on the free energy and its derived quantities. Using newly developed multiscale techniques to coarse grain the configuration space, we determined also these free energy contributions with a numerical error well below 1 meV/atom and were able to conclude this long standing debate: The contribution of **vacancies** in the free energy and the expansion coefficient is rather small, but still yields a considerable increase ( $\approx 0.1 k_B$ ) of the quasiharmonic result for the heat capacity. In contrast to this and to common expectations, the contribution of **explicit anharmonicities** to the heat capacity is always negative ( $\approx -0.1 k_B$ ) and are thus not able to explain the remaining discrepancy. For the expansion coefficients, the shift caused by their contribution (solid lines in Fig. 3) is also significant and ensures again that LDA and GGA describe the experiment equally well.

In the next step we extended the study to magnetic metals, as they are crucial for many engineering materials. In contrast to the previous two materials, the quasiharmonic approximation for bcc iron shows relatively large deviations

from the CALPHAD data already for the *ab initio* free energy, which become even more apparent in the heat capacity (dashed line in Fig. 4). The missing entropy contribution in this case is given by the **magnetic excitations**. Magnon energies, the relevant magnetic excitations at low temperatures, can be computed in DFT using the generalized Bloch theorem. Based on these calculations we have set up a model Hamiltonian (isotropic Heisenberg model), which allows to simulate the thermodynamics of the spin systems. Using classical methods for this purpose turned out to be insufficient and a sophisticated quantum-mechanical treatment was necessary. We have developed such a theory based on the random phase approximation [7] and afterwards combined magnetic, vibrational and electronic contributions to a complete free energy expression. Our *ab initio* derived heat capacity (solid line in Fig. 4) reproduces details of the experimental data for  $\alpha$  iron with a hitherto unachieved accuracy.

The development of *ab initio* approaches to predict the free energy of single phases is a crucial first step to our ultimate goal, the prediction of phase diagrams. Martensitic phase transitions, which are important in steels, can be nicely studied in several chemically ordered Heusler alloys, where they cause shape memory effects. As a final example of what this approach allows to study, we discuss the free energies of the three most relevant phases of  $Ni_2MnGa$ : The low temperature martensite, the austenite (the experimentally observed high temperature structure) and the pre-martensite



**Fig. 5:** *Ab initio* free energies for the most relevant phases in the magnetic shape memory alloy  $Ni_2MnGa$ . The martensite (green line) is taken as a reference; the austenite (red lines) and the pre-martensite (blue lines) are treated with (solid lines) and without (dashed lines) considering magnetic excitations.



(observed below 250 K). In Fig. 5 one can clearly see, that only the consideration of multiple free energy contributions yields the correct behaviour. The dashed lines correspond again to a calculation, where only vibrational and electronic contributions are taken into account. As a consequence, the austenite is unstable at all temperatures. Only when magnetic excitations are added to the free energy (solid lines), the whole sequence of structural phase transitions is accurately obtained [8].

In conclusion, we were able to systematically evaluate the predictive power of fully parameter free DFT calculations at finite temperatures. Based on extensive method developments, we were able to derive free energies with an extreme high numerical accuracy. Applying this approach on a wide range of materials, a hitherto not expected agreement with experiment has been found. Remaining deviations between present day DFT-functionals can be used as indications for the *ab initio* error bar. Even if these errors are for some temperatures/materials systems in the order of 10 meV/atom, the free energies can be used for deriving a detailed understanding of the thermodynamic properties. When making statements on physical processes, it is often more appropriate to study the second derivatives of the free energy, such as heat capacities and expansion coefficients, since small effects then become more pronounced. The ability of our approach to separately consider the various free energy contributions and in this way to systematically evaluate their physical relevance is a crucial advantage in this context. A decisive prerequisite for this purpose is the fact that we

have now all tools at hand to calculate the various free energy contributions (harmonic vibrations, quasiharmonic vibrations, non-linear Grüneisen parameters, explicit anharmonicities, electronic excitations, magnetic excitations, vacancies, self-interstitials) with an extremely high numerical accuracy ( $<1\text{meV/atom}$ ). These tools turned out to be sufficient for a faithful determination of phase transition temperatures in magnetic shape memory alloys with an accuracy well below 50 K. They are now applied to further technological relevant materials systems, for which experimental data are not yet available.

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# Hydrogen Embrittlement: An Atomistic Study of the HELP Mechanism

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The prospect of a H-based economy has resulted in renewed interest in the long-standing problem of H-induced embrittlement of metals, which can be loosely defined as the premature failure of metals under the influence of H. The effect has been observed in fcc, bcc, as well as in hcp metals, but is particularly pronounced in high strength steels [1].

Various mechanisms have been proposed to account for the observed effect, including (i) stress-induced hydride formation, (ii) H-enhanced decohesion (HEDE), (iii) H-induced vacancy cluster formation, (iv) H-induced cracking and (v) H-enhanced local plasticity (HELP) [1]. However, it is generally agreed that none of these mechanisms will account for the H embrittlement on its own, but rather a combination of some of these mechanisms is expected to underlie the observed failure, depending on the nature of the material.

Experimental evidence based on high resolution fractography, as well as *in-situ* TEM studies strongly suggest a distinctly plastic nature of H induced fracture [1,2], which led to the development of the HELP mechanism that correlates the ostensibly conflicting observations of increased microscopic plasticity and macroscopic embrittlement. The fundamental assumption of this theory is that the increased plasticity is localised to regions of high H content, resulting in localised plastic fracture. On an atomistic level this mechanism is based on the assertion that H accumulates in the tensile strain field of dislocations, due to the increased volume of the interstitial cavities in that region. Using continuum elasticity theory the accumulation of H has been shown to shield the stress fields around dislocations [2], which results in a weakening of the repulsive dislocation-dislocation interaction and hence allows for increased dislocation mobility in regions of high H concentrations, such as crack tips and notches. The localised plasticity postulated by the HELP mechanism is thus induced by the localised distribution of the H atoms.

The atomistic mechanism underlying the theory has not been validated so-far. In particular, the distribution of H around the dislocations underlying the continuum model is based on the elastic interaction between the strain field of an edge type dislocation as predicted by linear elasticity theory and the strain field around isolated H interstitials within the host lattice, while the important H-H interaction, which can significantly

affect the interstitial solubility [3], is disregarded. It was therefore the aim of this study to investigate the effect of H on the stress fields of edge dislocations in metals on an atomistic level, using the NiH system as a model, which has previously been found to exhibit the hallmark of localised plastic fracture [2] and for which well established semiempirical potentials are available.

**Methodological aspects.** The HELP mechanism is a challenging problem since it involves interactions at different length scales: The accurate description of the short ranged, chemical metal-H and H-H interactions requires in principle an *ab initio* electronic structure calculation, while the long-ranged strain interaction necessitates system sizes that render such calculations computationally unfeasible and can therefore only be described by computationally less demanding semiempirical potentials, such as embedded atom (EAM) type potentials. In addition, the H distribution within the dislocation system involves a huge configurational space, which renders the distribution of the interstitial H atoms by Monte Carlo (MC) simulations computationally demanding, even for the computationally efficient EAM potentials. In order to facilitate the sampling of this huge configurational space a lattice gas (LG) Hamiltonian, describing the energy of an interstitial H atom as a function of the volume of the interstitial cavity and the number of H atoms in first and second nearest interstitial sites, was developed. Hence, a hierarchical approach was adopted to accommodate the scale-transcending nature of the physical phenomena underlying the HELP mechanism: H-H and metal-H interaction parameters determined by density functional theory (DFT) calculations were used to validate the corresponding parameters calculated using a binary EAM potential, which in turn was used to parametrise the LG Hamiltonian.

**Results.** DFT calculations at the level of the spin-polarised generalised gradient approximation to the exchange-correlation functional [4-6] were performed to determine the H-H and Ni-H interactions, by calculating the stability of interstitial H clusters within the Ni matrix, represented by a  $2 \times 2 \times 2$  supercell. H atoms were successively placed in the twelve nearest neighbour octahedral (O) sites around a central O site and the energies of the twelve clusters thus obtained were determined by performing ionic relaxations, while constraining the cell dimensions to the equilibrium lattice constant of Ni. For clusters

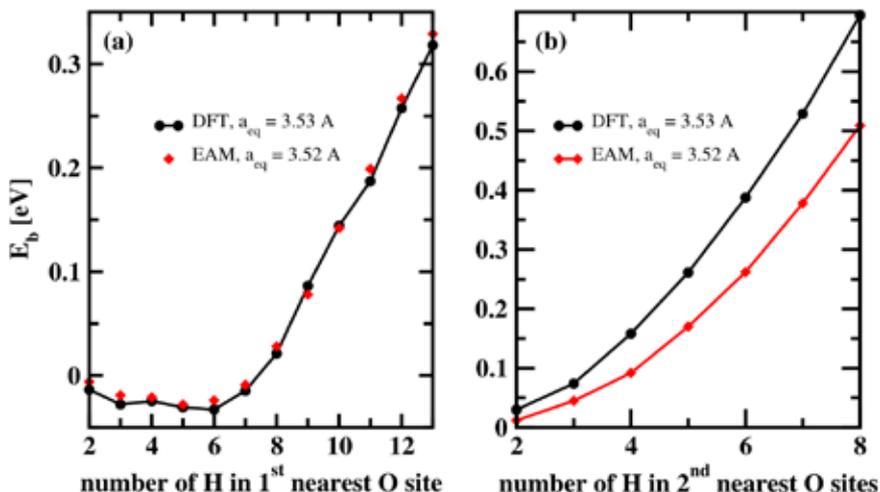


Fig. 1: Binding energies of clusters of interstitial H atoms in a  $2 \times 2 \times 2$  supercell of Ni as a function of the number of H in (a) 1<sup>st</sup> and (b) 2<sup>nd</sup> nearest neighbour O sites.

consisting of up to seven interstitial H atoms, negative binding energies were found (see Fig. 1(a)), indicating their stability with respect to a random distribution of the interstitial atoms, which is attributed to a strong, attractive chemical interaction that compensates for the repulsive strain interaction between adjacent interstitials. Next the H-H interaction between second nearest interstitial sites was considered by determining the energy of the eight cluster structures obtained by successively occupying all O sites within the  $2 \times 2 \times 2$  supercell that are separated from any other interstitial H atom by at least one conventional lattice constant (2<sup>nd</sup> nearest neighbour distance). A repulsive interaction was found irrespective of the cluster size (see Fig. 1(b)), indicating a significantly weakened chemical interaction as compared to the next nearest neighbour interaction.

The accuracy of the binary EAM potential for the NiH system used in this study [7,8] was validated by comparing the corresponding H-H interaction parameters to the results obtained by the DFT calculations. As shown in Figs. 1(a) and (b), a surprisingly good correspondence is found. In particular, the 1<sup>st</sup> nearest neighbour H-H interaction energies obtained from the DFT calculations are almost perfectly reproduced by the corresponding EAM results, while the repulsive interaction between H in 2<sup>nd</sup> nearest interstitial sites is somewhat underestimated. The close match between the *ab initio* and EAM results suggested that the H distribution can be determined using the EAM potential, without reverting to computationally demanding DFT calculations.

The LG Hamiltonian was developed by interpolating the dependence of the interstitial H energy on the volume of the interstitial cavity and the number of H atoms in 1<sup>st</sup> and 2<sup>nd</sup> nearest interstitial sites, as determined using the EAM potential. The dependence on the interstitial volume was established by determining the energy of an isolated H atom at every interstitial site within the strain field of an  $\{111\} a_0/2$  [110] edge type dislocation, yielding a linear energy-volume relationship. The H-H interaction parameters, on the other hand, were determined

by parametrising cluster formation energies within a  $7 \times 7 \times 7$  supercell, as the strain energy of the interstitial H atoms was overestimated in the  $2 \times 2 \times 2$  supercell considered before, due to the interaction with spurious images of the hydrogens in neighbouring supercells. The H-H interaction parameters are qualitatively changed by considering the larger supercell, as shown in Fig. 2. Hence the interaction between H atoms in 1<sup>st</sup> nearest neighbour interstitial sites is found to be attractive for all cluster sizes considered (containing up to 13 H atoms) and the repulsive interaction between H atoms in 2<sup>nd</sup> nearest interstitial sites is significantly weakened.

Interstitial H atoms were now distributed around a single  $\{111\} a_0/2$  [110] edge type dislocation by performing Metropolis MC simulations using the LG Hamiltonian in the grand canonical ensemble with the chemical potential ( $\mu_H$ ) chosen to realise a bulk concentration of 0.25 at. %. The target concentration was chosen to match the expected bulk concentration, as determined from experimental heat

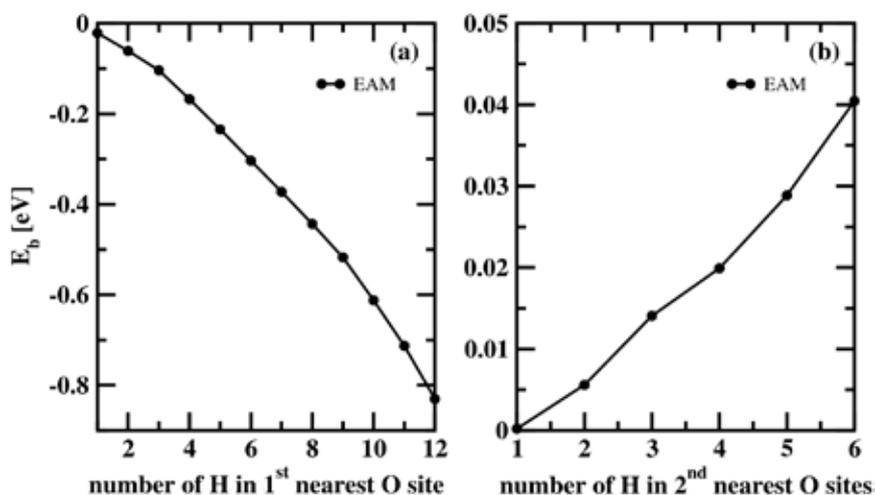
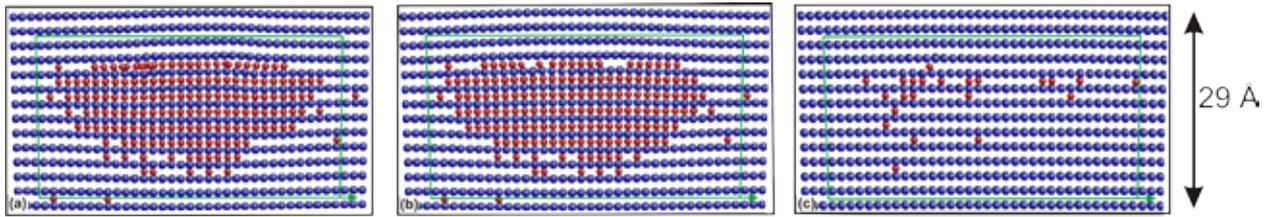


Fig. 2: Binding energies of clusters of interstitial H atoms in a  $7 \times 7 \times 7$  supercell of Ni as a function of the number of H in (a) 1<sup>st</sup> and (b) 2<sup>nd</sup> nearest neighbour O sites.



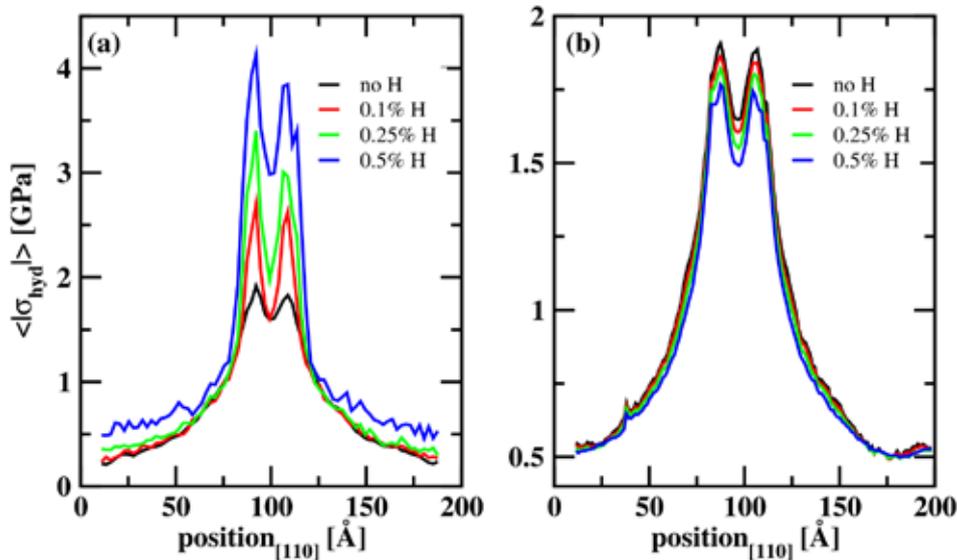
**Fig. 3:** *H* distribution around a single  $\{111\} a_0/2 [110]$  edge type dislocation in Ni determined using (a) the full LG Hamiltonian, (b) the EAM potential and (c) the truncated LG Hamiltonian disregarding the H-H interactions. Green lines show the Burgers circuits around the dislocation.

of solution data [9]. In order to check the accuracy of the LG parametrisation, the obtained distribution was compared to a corresponding distribution determined using the EAM potential. As shown in Fig. 3(a) and (b), the LG parametrisation describes the NiH system sufficiently well to allow for realistic H distributions. While the observed accumulation of H atoms within the tensile strain field around the dislocation was expected, the density of the distribution of almost 100 at. % along the dislocation line was surprising and suggested the formation of a hydride phase. The observed hydride formation along the dislocation line could be attributed to the strength of the H-H interaction: Redistributing the H atoms within the dislocation system using a truncated LG Hamiltonian (H-H interaction parameters turned off) yielded a significantly more loose distribution (see Fig. 3(c)).

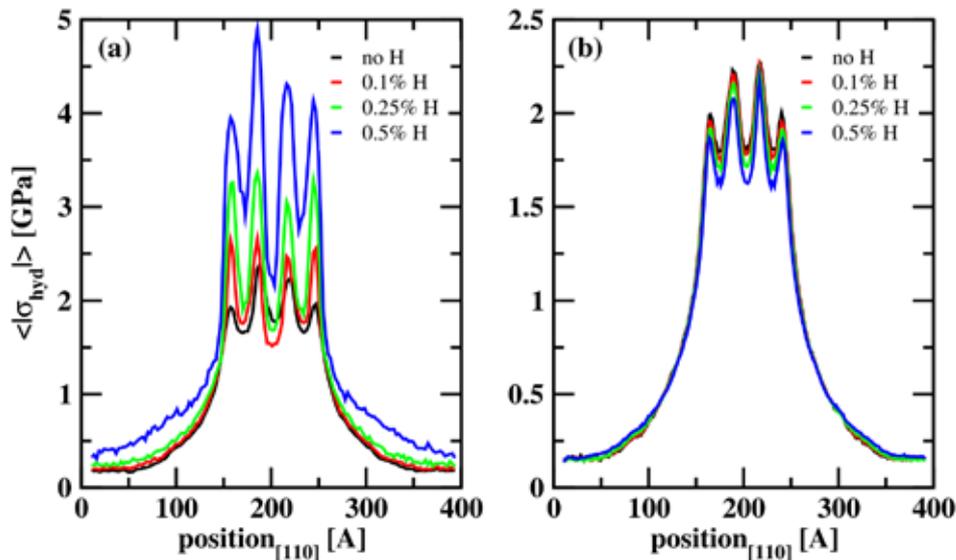
The effect of the hydride formation on the stress field around an edge dislocation was now investigated by determining the stress around a single  $\{111\} a_0/2 [110]$  edge dislocation. We considered two cases: the presence of the dense H distribution and the presence of the dilute H distribution using the full and truncated LG Hamiltonian, respectively. As shown in Fig. 4(a) and (b) two qualitatively different results

were observed. The hydrostatic stress around the dislocation in the presence of the hydride phase along the dislocation core is significantly increased as the target H concentration ( $\mu_H$ ) is increased, reflecting the mismatch in the equilibrium lattice parameters of the hydride (3.73 Å) and the bulk Ni phase (3.52 Å). Conversely, the dilute H distribution obtained by disregarding the H-H interaction term in the LG Hamiltonian, results in a somewhat decreased hydrostatic stress field around the dislocation. This is attributed to an increased shielding effect resulting from the interaction of the dilatational strain field of isolated interstitial H atoms and the tensile strain field of the dislocation, as predicted by the original formulation of the HELP mechanism. It is emphasised that the inclusion of the H-H interaction parameters for the distribution of H results in an inverted effect of the interstitial H atoms on the stress field around an isolated edge type dislocation.

Next the effect of H on the dislocation-dislocation interaction was investigated by determining the hydrostatic stress field around two parallel  $\{111\} a_0/2 [110]$  edge type dislocations in the presence of H. The formation of a hydride phase along the dislocation line due to the inclusion of the



**Fig. 4:** Planar average of the absolute value of the hydrostatic stress around a single  $\{111\} a_0/2 [110]$  edge type dislocation in Ni based on H distributions obtained using (a) the full and (b) the truncated LG Hamiltonian.



**Fig. 5:** Planar average of the absolute value of the hydrostatic stress around two parallel  $\{111\} a_0/2 [110]$  edge type dislocation in Ni based on H distributions obtained using (a) the full and (b) the truncated LG Hamiltonian.

H-H interaction parameters for the distribution of H yielded a surprising weakening of the stress field in between the dislocations for the lower H concentrations considered, although the increased stress field in the direct vicinity of the dislocation cores, described above, was reproduced (see Fig. 5 (a)). The observed weakening of the stress field in the region between the dislocations is attributed to a more efficient screening of the stress field around the dislocation, which results from the hardening of the material in the vicinity of the dislocation core, due to the hydride formation. Thus the formation of the hydride phase along the dislocation line has two competing effects on the stress field: It induces significant misfit stress and simultaneously results in a faster decay of the stress field around the dislocation. For the system studied here, the faster decay of the stress field within the hydride phase dominates in the case of the lower H concentrations considered, while for higher concentrations the misfit stress determines the net effect due to the large spatial extent of the hydride precipitate. The stress field around the two dislocations in the presence of the commonly assumed, but unstable dilute H distribution reproduces the result for a single dislocation: The hydrostatic stress field in between the two dislocations is (somewhat) weakened as the H concentration is increased (see Fig 5 (b)).

**Conclusions.** Our results highlight the importance of H-H interactions on dislocation-dislocation interactions in fcc materials. In the limit of no or very weak H-H interactions, a dilute H distribution is formed, resulting in a (minor) weakening of the dislocation-dislocation interaction, as predicted by the continuum calculations underlying the original formulation of the HELP mechanism. However, with increasing strength of the H-H interaction a denser H distribution is formed, giving rise to a local hydride

phase along the dislocation line. The concentration dependent effect of the hydride formation on the dislocation-dislocation interaction was explained in terms of a delicate balance between (i) the misfit stress resulting from the different lattice constants and (ii) the relative hardness of the bulk and the hydride phase.

We therefore conclude that the HELP mechanism is viable in principle, but more material specific than previously assumed. In particular, the formation of a hydride phase along the dislocation line in metals in which the interstitial H-H interaction is strong, can result both in increased and decreased dislocation-dislocation interactions depending on (i) the lattice constant mismatch between the hydride and the metal, (ii) the relative hardness of the two phases and (iii) the H-concentration.

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## First Principles Predictions of Stacking Fault Properties in High-Mn Steels

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High-Mn-steels are excellent candidates for the next generation of light-weight high-strength steels due to their exceptional mechanical characteristics. The mechanical properties of such steels sensitively depend on their microstructure. The stacking faults that occur in the austenitic phase of high-Mn steels are of particular importance since their energies define which plasticity mechanism (twinning induced or transformation induced plasticity, TWIP or TRIP) prevail in the steel [1]. Up to now the theoretical understanding of such defects was based on a combination of the regular and subregular solution models and the Olson-Cohen approach [2]. The parameters entering these models are either derived from explicit measurements of the stacking fault energies (SFEs), or from empirical relations for the Gibbs free energy  $G$  of *fcc* and *hcp* phases of the material [3]. Such an approach relies on the continuum description of individual *fcc* and *hcp* phases and does not account for the atomistic structure of the stacking faults. However, in order to effectively explore chemical trends, to deliver parameters for phenomenological models, and to identify new routes to optimize high-Mn-steels an atomistic understanding of such structural defects is decisive. The ultimate goal of the current research is the development of a generalized first principle theoretical framework that allows an accurate temperature- and composition-dependent description of the stacking fault properties in various types of steels, and the application of the method to the particular case of high-Mn steels. The framework is based on density functional theory (DFT), which is based on the explicit description of the chemical bonding between atoms and is an established tool for the deriving material properties. Since Fe and Mn chemical species are the two dominating types of atoms in realistic high-Mn-steels, we first start with the description of the stacking faults in the FeMn-alloy. The actual steels can be obtained by admixing small concentration of other chemical elements like, e.g., carbon, Si, etc. substitutionally or interstitially in this host structure.

Austenitic FeMn-alloys are ordered antiferromagnetically (aFM), with the Néel temperature being ~350-400 K at Mn contents larger than 20 atomic percents [5]. Information regarding the actual atomic and magnetic structure, i.e. whether there exist short-

range or long-range order, is lacking. To calculate the stacking fault energies we, therefore, first had to identify the actual atomic arrangement as function of temperature and chemical composition.

The existing first principle calculations of the SFE can be divided into two classes: They are either performed for materials at their ground state at  $T=0$  K (see, e.g. Ref. [6-8]), i.e. completely neglecting the temperature dependence of the SFE, or temperature-dependent calculations for the paramagnetic disordered phase [9]. The approach employed in the latter calculations is, however, not applicable to steels where atomic relaxations are significant. For example, it cannot account for changes in the structure that may occur at the stacking faults, and is limited to high temperatures where no ordering exists. In order to calculate the SFE in FeMn-alloys over a wide range of temperatures and compositions it is necessary, therefore, to develop a new methodology that is able to include (i) the change of the atomic (magnetic) structure as function of temperature and composition, (ii) provides an effective method for evaluating the SFE for a given representative structure, and (iii) is able to accurately account for the temperature effects. Below we present the first steps towards constructing such an approach on the example of FeMn-alloys.

In order to estimate the importance of the magnetic interactions we first switched them off and treated the alloy as nonmagnetic. The only structural unknown is then the chemical structure. To identify it over the complete range of Mn compositions we considered two distinct extreme cases: (i) the low temperature limit, where chemically ordered structures are energetically most stable, and (ii) the high temperature limit, where entropy contributions are large and completely destroy any ordering in the system, resulting in the chemically disordered alloy.

The ground-state atomic configurations corresponding to the low temperature limit have been identified by the cluster-expansion method [10], which allows an effective mapping of the first principles DFT calculations onto a model Hamiltonian. The model Hamiltonian has then been used for calculating the total energies of numerous crystal structures with different arrangements of Fe and Mn atomic species.

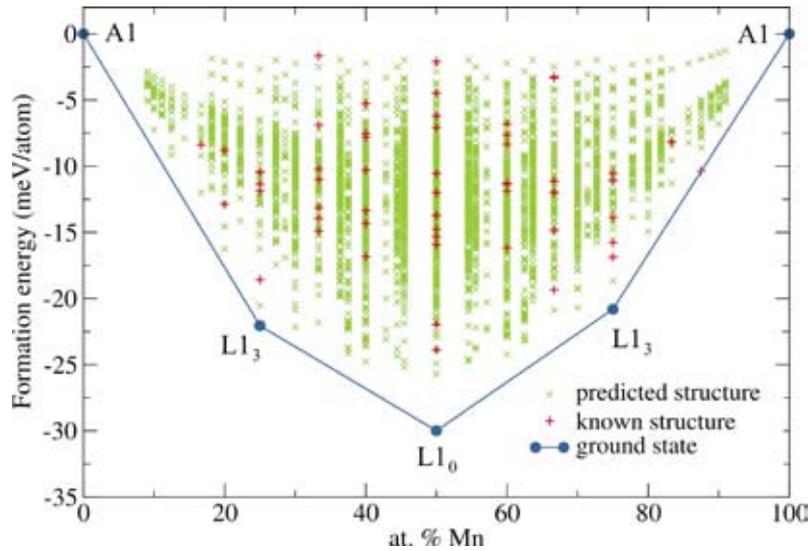


Fig. 1: Ground-state structures of nonmagnetic austenitic FeMn-alloys identified by the cluster expansion method.

Employing this method we have considered more than 5000 distinct atomic configurations without loss of accuracy, and reliably identified the true ground state structures (A1, L1<sub>3</sub>, L1<sub>0</sub>) of FeMn-alloy over the entire range of Mn concentrations (see Fig. 1). In a second step we used these ground state structures to simulate the so-called generalized stacking fault energy ( $\gamma$ -surface). It is obtained by shifting two half crystals of the austenitic material with respect to each other along the (111) glide plane by a given displacement vector, and calculating the SFE  $\Gamma$  as an excess energy that is conserved in the defect area:

$$\Gamma = (G^{\text{def}} - G^{\text{ideal}}) / A_{\text{int}} \quad (1)$$

Here,  $G^{\text{def}}$  is the free energy of a crystal containing the defect,  $G^{\text{ideal}}$  is that of the ideal *fcc* crystal, and  $A_{\text{int}}$  is the area over which the stacking fault extends in the (111) plane. The positions of the global minima on the calculated  $\gamma$ -surface correspond to intrinsic stacking faults which realize the *fcc/hcp* transition in the crystal due to slips. A comparison with available empirical data (see Fig. 2) indicates that the calculated SFE for nonmagnetic ordered alloys does not reproduce the available empirical data: while the empirical SFE decreases with increasing Mn content at low Mn concentrations, goes through a minimum and then increases with higher Mn-concentrations, the calculated SFE monotonously increases with increasing Mn content. Further, the predicted SFE are largely negative (below  $\sim -300$  mJ/m<sup>2</sup>) while the extrapolation of the empirical data to  $T=0$  K results in values of the SFE not lower than  $-40$  mJ/m<sup>2</sup>. This large discrepancy indicates that magnetic interactions and/or chemical ordering play a crucial role in FeMn-alloys and have to be taken into account in the simulations.

In order to identify which of the two remaining mechanisms is responsible for the observed discrepancy we calculated in a next step intrinsic SFES

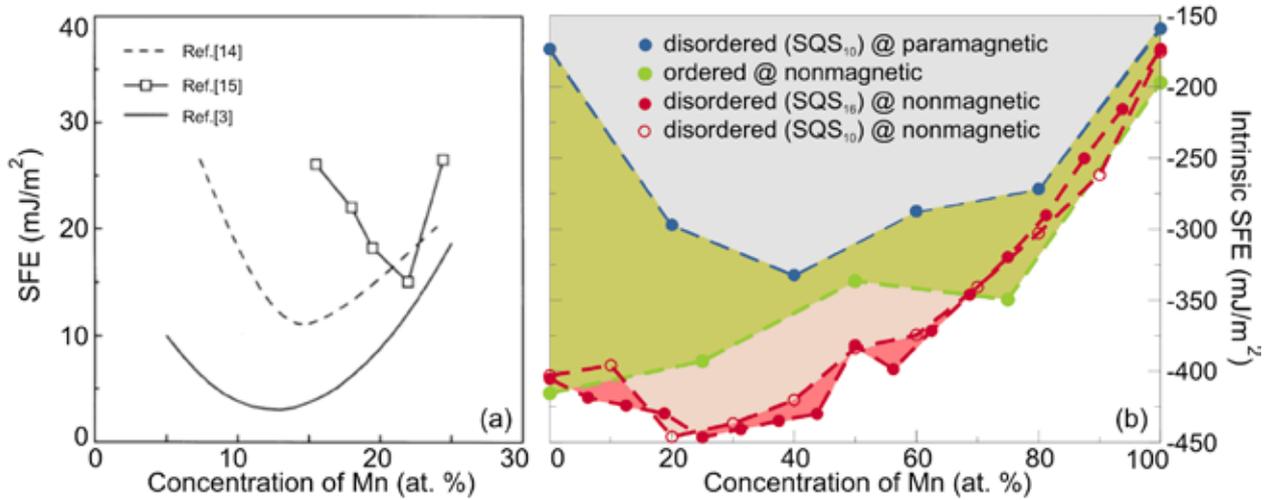
for the disordered (high temperature) phase of nonmagnetic FeMn-alloys. To represent random alloys we utilized the concept of special quasirandom structures (SQS) [11]. This method guarantees the best possible description of an ideal random structure for a given number of atoms in a periodically repeated supercell, and allows to properly include atomic relaxations. To calculate the SFE for random alloys within this approach, we had to reduce the computational complexity and employed the axial next-nearest-neighbor Ising (ANNNI) model [12]. This model had already been successfully applied to austenitic stainless steels [9]. The ANNNI model describes the actual intrinsic stacking fault from a series expansion of different infinitely repeated stacking sequences. In the first order (Eq. (2)) only the energies of the *fcc* and *hcp* stacking sequences are considered, while higher-order expansions incorporate more sophisticated phases, like, e.g. the double *hcp* phase (Eq. (3)):

$$\Gamma^1 = 2 (G^{\text{hcp}} - G^{\text{fcc}}) / A_{\text{int}} \quad (2)$$

$$\Gamma^2 = (G^{\text{hcp}} + 2G^{\text{dhcp}} - 3G^{\text{fcc}}) / A_{\text{int}} \quad (3)$$

The results for the intrinsic SFE (ISFE) calculated for disordered nonmagnetic FeMn-alloys are shown in Fig. 2(b). In contrast to the low temperature case where only ordered structures have been assumed, the qualitative agreement with the empirical data is improved: The SFE no longer monotonously increase with the Mn concentration, but correctly reproduce the minimum of the SFE at medium Mn content ( $c_{\text{Mn}} \approx 25\%$ ). Quantitatively, however, the agreement is less satisfactory and is similar to a case of the ordered nonmagnetic alloy.

Because the type of the antiferromagnetic ordering (e.g., layer-wise aFM, double layer aFM, spin-spiral state), as well as the chemical order-disorder transition temperature is not known from experiment,



**Fig. 2:** (a) Empirical data for SFE austenitic FeMn-alloys as a function of Mn content at room temperature (b) Summary of the corresponding calculated intrinsic SFE at  $T=0$  K. The high temperature magnetic phase (paramagnetic chemically disordered alloys), as well as the nonmagnetic phase at low temperature (chemically ordered alloys) and at high temperature limit (chemically disordered alloys) are shown. The results for the chemically ordered phase are obtained by explicitly calculating the  $\gamma$ -surface. For the various disordered phases - the 1<sup>st</sup>-order ANNNI model (see text) has been employed.

we consider first the paramagnetic phase of the FeMn-alloys and assume that no chemical order exists in the system. Such a paramagnetic chemically disordered alloy corresponds then to the high temperature state of the crystal. To simulate such a scenario we have employed a similar approach as used for nonmagnetic disordered alloys, i.e. a combination of the SQS and the ANNNI model: the paramagnetic state of the system was simulated with quasiquaternary alloys  $\text{Fe}_{x/2}^{\uparrow}\text{Fe}_{x/2}^{\downarrow}\text{Mn}_{(1-x)/2}^{\uparrow}\text{Mn}_{(1-x)/2}^{\downarrow}$  where a collinear magnetic structure is assumed and  $\uparrow, \downarrow$  correspond to up and down spin channels of the system. This approach is formally equivalent to the disordered local moment (DLM) model, that is known to provide a reliable description of the paramagnetic state of the crystal [9]. The results of the corresponding calculations are shown in Fig. 2(b) by the blue dots. They clearly indicate that magnetic interactions indeed play an essential role in FeMn-alloys and cannot be disregarded. Specifically, the ISFE are increased for all compositions when compared with the corresponding nonmagnetic structures, with the most pronounced magnitude of the increase at low Mn-content. Further, the experimentally observed presence of the SFE minimum at Mn-concentrations less than 50% is reproduced. Still, although the quantitative agreement between the first principles and empirical data has been significantly improved by accurately taking into account magnetism and chemical disorder, the present level of accuracy is still not sufficient for qualitative prediction of the plastic properties of FeMn-alloys, for which errors in the order of several  $\text{mJ/m}^2$  may become important.

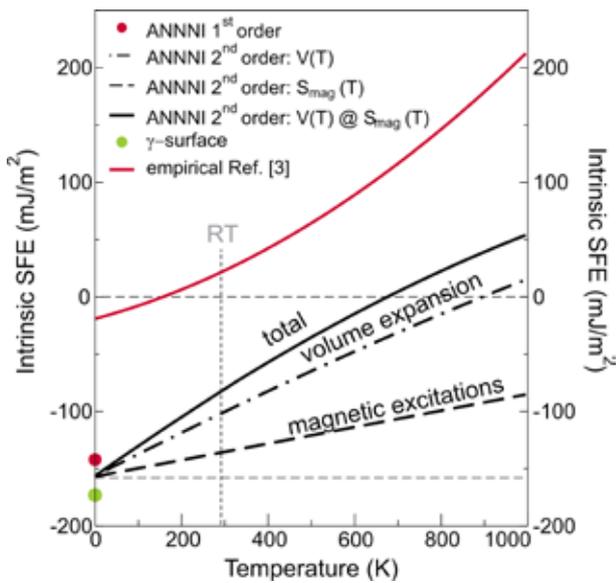
To identify the source of the remaining discrepancy we restrict here on the case of pure *fcc* Fe, where no complication due to unknown chemical order

exist. We note first that the calculated SFE of the paramagnetic disordered phase corresponds to  $T=0$  K. In reality, however, this state is realized only at elevated temperatures. For an adequate comparison, therefore, it is necessary to account for the temperature dependence of the SFE. In the framework of the ANNNI model the problem substantially simplifies and requires only a description of the temperature-dependent free energy  $G(T)$  of bulk crystals with different stacking sequences. Although it is principally possible to calculate  $G(T)$  explicitly (see, e.g., p. 205), we employ here a more approximate treatment of the temperature effects. Specifically, we explicitly include the volume change with temperature by extracting the thermal linear expansion coefficient from experimental data and extrapolating it to low temperature [13]. We further include the effects of magnetic entropy via the mean-field expression:

$$S^{\text{mag}}(V) = \sum k_B \ln(2S_i(V)+1) \quad (4)$$

where  $S_i(V)$  is the volume dependent spin quantum number of the atom  $i$ , which can be related to the corresponding localized atomic magnetic moment  $m_i(V) = 2S_i(V)$  [9]. The contribution due to vibrational entropy is not included. The obtained estimate of the free energy  $G(T) = E(V(T)) - TS^{\text{mag}}(V(T))$  corresponds to the upper limit for both the magnetic entropy and the thermal volume expansion.

In Fig. 3 we show the comparison of the temperature-dependent theoretical calculations for the SFE in paramagnetic *fcc* Fe with an empirical relation for the SFE in FeMn-alloys at the limiting case of zero manganese content. The calculated SFE sensitively depend on the temperature, and, in agreement with the empirical curve, correctly predicts a nonlinear



**Fig. 3:** Temperature dependence of the intrinsic stacking fault energy in paramagnetic fcc Fe obtained when the thermal volume expansion and the contribution of the magnetic excitations are included. The empirical dependence is taken from Ref. [3].

increase of the SFE with temperature. However, because the magnitude of the SFE at  $T=0$  K in our calculations is  $\sim 130$  mJ/m<sup>2</sup> smaller than the corresponding empirical value, the temperature effects are not strong enough to fully reproduce the experiment. Our calculations further show (Fig. 3), that although the magnetic excitations are significant, the thermal volume expansion dominates the increase of the SFE with increasing temperature. Such pronounced volume dependence is unexpected and was assumed in previous studies to be negligible [9]. We note, that in actual alloys the volume might change not only due to an increase of the temperature, but might occur due to adding other species like e.g. Carbon, applying external strain/stress, or in the vicinity of local strain fields.

In summary, we have presented a generalized first principle approach for calculation of the stacking fault energy in realistic steels. The method is based on a multiphysics combination of approaches from alloy physics, microstructure physics, physics of magnetism, and thermodynamics. We have briefly highlighted the method for calculating the SFE for high- and low-temperature phases of nonmagnetic binary alloys, as well as a method for calculating the high-temperature phase of the magnetic binary alloys. For the considered case of FeMn-alloys our results showed a hitherto unexpected conclusion: In contrast to conventional wisdom that stacking fault related plasticity mechanisms can be described by a single value for the SFE, the results clearly show that

the volume-dependence of the stacking fault energy should be included. An important consequence is that the prevailing plasticity mechanisms (TRIP/TWIP) can even change upon deformation of FeMn-alloys. These results indicate that a careful revision of the available experimental data should be made. For this a set of reference experiments on SFE in single-crystal FeMn-alloy will be performed in collaboration with the MU department (Prof. Raabe). We finally note that the presented approach is not limited to binary alloys, and allows a straightforward generalization to multicomponent systems, including lattice mismatch alloys where atomic relaxations become essential. Further development of the model towards identification of the stacking fault properties at the intermediate temperature range where chemical and/or magnetic ordering exist, as well as a more accurate inclusion of the various temperature contributions to the SFE, is still required and is subject of ongoing research.

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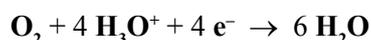
## Electrochemical Oxygen Reduction Mechanism in Aqueous Media

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Department of Interface Chemistry and Surface Engineering

Oxygen is a key factor in many corrosion processes. The electrochemical reduction of oxygen at a metal surface is the cathodic partial reaction and driving force for corrosion leading to metal dissolution and/or oxide formation. On the other hand, electrocatalytic oxygen reduction in fuel cells, an environmentally friendly, portable power source, is intensely studied with the aim of improving the kinetics and reducing the overpotential that limits the energy efficiency.

The electrochemical reduction of oxygen is a complex multi-step process. In the reaction



overall 4 protons and 4 electrons are transferred. There also is a two electron reduction leading to hydrogen peroxide (**HOOH**). The mechanism is controversial and was recently discussed, e.g., at the 140<sup>th</sup> Faraday Discussions "Electrocatalysis - Theory and Experiment at the Interface" [1], and in a dedicated issue of PCCP [2], with several experimental as well as theoretical contributions. The reaction mechanism in electrolyte is different from surface catalysis of gas phase oxidations, which also has been studied extensively (see e.g., [3]). Solvation by water stabilizes charged intermediates, making them energetically competitive. Furthermore, hydrogen-bonding and proton transfers increase the electron affinity of the reactants. For this reason the reaction mechanism may vary, depending on the reaction conditions, in particular, the pH of the solution, the electrode potential and the nature of the electrode surface.

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In this theoretical study, the reaction mechanism of the electrochemical oxygen reduction in aqueous solution was analyzed, based on the assumption that the elemental reaction steps correspond to either single electron transfers or single proton transfers. For this purpose, all intermediates that may be generated from oxygen (**O<sub>2</sub>**) by sequentially adding electrons and protons in arbitrary order were considered as shown in Tab. 1. Species with charges up to  $\pm 2$  were included.

For a realistic description, the effects of the solvent, of the pH and the electrode potential are essential. The accuracy of several approaches for simulation of solvent effects was tested using well characterized reactions. The best performing solvation model was applied to the oxygen reduction reaction.

**Computational methods.** The calculations were done with the Gaussian03 program [4] using the B3PW91/aug-cc-pVDZ hybrid density functional and basis set. Relative energies calculated at this level agree with CCSD(T)/aug-cc-pVQZ results within  $\pm 0.01$  eV for unsolvated and  $\pm 0.1$  eV for PCM solvated intermediates. Augmenting the basis set with diffuse functions (aug) is essential, but DFT calculations give converged results already for a double zeta (DZ) basis, while CCSD(T) requires a quadruple zeta (QZ) basis. The cavities for PCM solvation were defined by the UFF model with spheres on all atoms including hydrogens. Non-electrostatic contributions were neglected as they cancel out within  $\pm 0.01$  eV. Free energies were calculated by adding zero-point and thermal corrections based on calculated harmonic frequencies. The pH was included via the concentration dependent term  $-RT \ln(C/C_0)$ .

		+ e <sup>-</sup>	+ 2 e <sup>-</sup>	+ 4 e <sup>-</sup>	+ 4 e <sup>-</sup>
	<b>O<sub>2</sub></b>	<b>O<sub>2</sub><sup>•-</sup></b>	<b>O<sub>2</sub><sup>2-</sup></b>		
+ H <sup>+</sup>	<b>HOO<sup>+</sup></b>	<b>HOO<sup>•</sup></b>	<b>HOO<sup>-</sup></b>	<b>O<sup>•-</sup> + OH<sup>-</sup></b>	
+ 2 H <sup>+</sup>	<b>HOOH<sup>2+</sup></b>	<b>HOOH<sup>•+</sup></b>	<b>HOOH</b>	<b>O<sup>•-</sup> + H<sub>2</sub>O</b> <b>OH<sup>•</sup> + OH<sup>-</sup></b>	<b>2 OH<sup>-</sup></b>
+ 3 H <sup>+</sup>		<b>H<sub>2</sub>OOH<sup>2+</sup></b>	<b>H<sub>2</sub>OOH<sup>+</sup></b>	<b>OH<sup>•</sup> + H<sub>2</sub>O</b>	<b>OH<sup>-</sup> + H<sub>2</sub>O</b>
+ 4 H <sup>+</sup>				<b>H<sub>2</sub>O<sup>•+</sup> + H<sub>2</sub>O</b>	<b>2 H<sub>2</sub>O</b>

**Tab. 1:** Scheme of intermediates considered for the electrochemical oxygen reduction reaction. Horizontal steps correspond to one electron reductions, vertical steps to proton transfers.

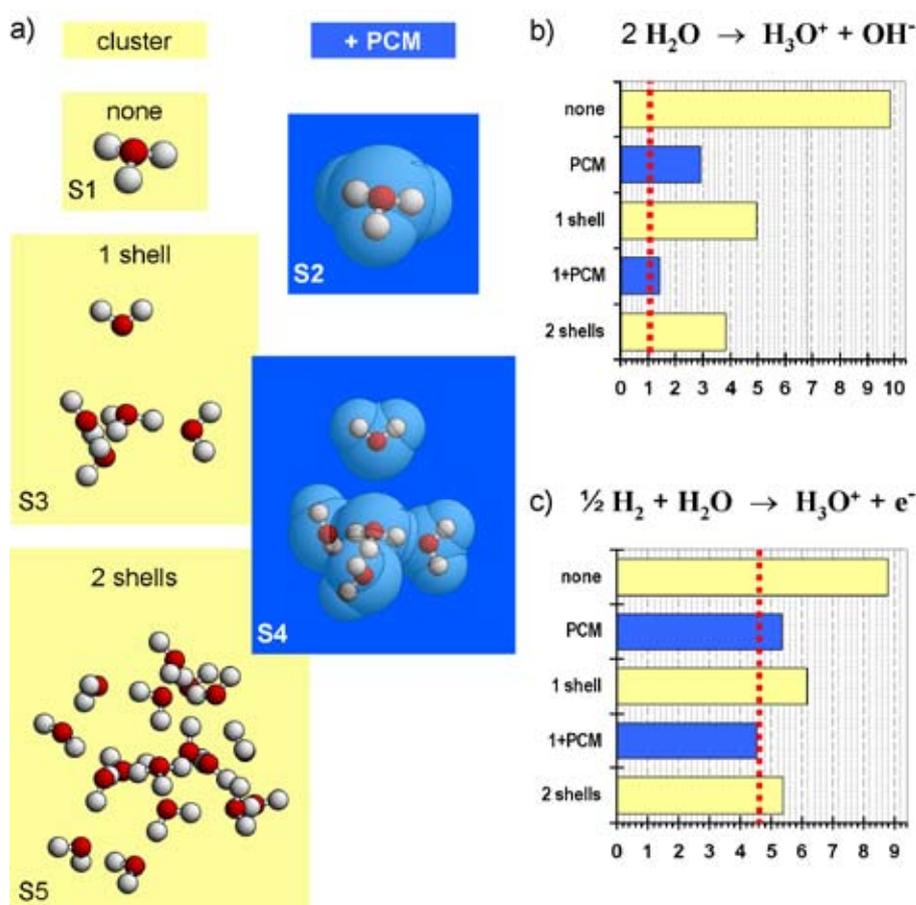


Fig. 1: The five solvation models illustrated for the solute  $\text{H}_3\text{O}^+$  and their performance.

### Hierarchy of Solvation Models:

**S1, gas phase:** For comparison the unsolvated solute species were calculated.

**S2, PCM:** The electrostatic stabilization of a charged or polar species in a polar solvent may be calculated using the implicit solvation model of a polarizable continuum (PCM) [5]. The solutes are placed in a cavity inside a dielectric medium with  $\epsilon = 78.39$  simulating bulk water and the charge distribution is recalculated in the presence of the reaction field of the continuum until self-consistency is reached. The PCM model efficiently estimates the long-range electrostatic stabilization, however neglects specific short-range interactions as, e.g., hydrogen bonds.

**S3, one shell of explicit water molecules:** Water molecules, and ions like  $\text{OH}^-$  and  $\text{H}_3\text{O}^+$  may accept/donate four hydrogen bonds forming an approximately tetrahedral coordination in their first hydration shell. Molecules like  $\text{O}_2$ , hydrogen peroxide ( $\text{HOOH}$ ), and similar species may accept/donate six hydrogen bonds. Thus six water molecules were used for the first solvation shell coordinating all lone pairs and hydrogens. The structure of the clusters constructed from solute and first solvation shell has been optimized starting from an idealized structure

and imposing tetrahedral angles between the oxygen atoms of the solvent molecules to enforce an even distribution of the solvent molecules and a bulk water like structure.

**S4, one shell of explicit water in combination with PCM:** The cluster of solute plus first solvation shell (as in S3) was placed in a cavity of polarizable continuum. The structures were reoptimized resulting in minor changes.

**S5, two shells of explicit water:** Each water molecule of the first solvation shell was surrounded by three water molecules of the second solvation shell (a fourth hydrogen bond connects the 1st-shell water molecule to the solute). The second shell water molecules form hydrogen bonds with neighbouring molecules in the second shell. This leads to 5-rings of cyclic hydrogen bonds that stabilize the structure of the cluster allowing full optimization of the clusters  $\text{H}_3\text{O}^+ \cdot 16 \text{H}_2\text{O}$ ,  $\text{OH}^- \cdot 16 \text{H}_2\text{O}$ , and  $\text{H}_3\text{O}^{+*} \cdot 16 \text{H}_2\text{O}$ .

**Validation:** The performance of the five solvation models illustrated in Fig. 1a was tested using the water autoprotolysis reaction,



and the reaction of the standard hydrogen electrode,

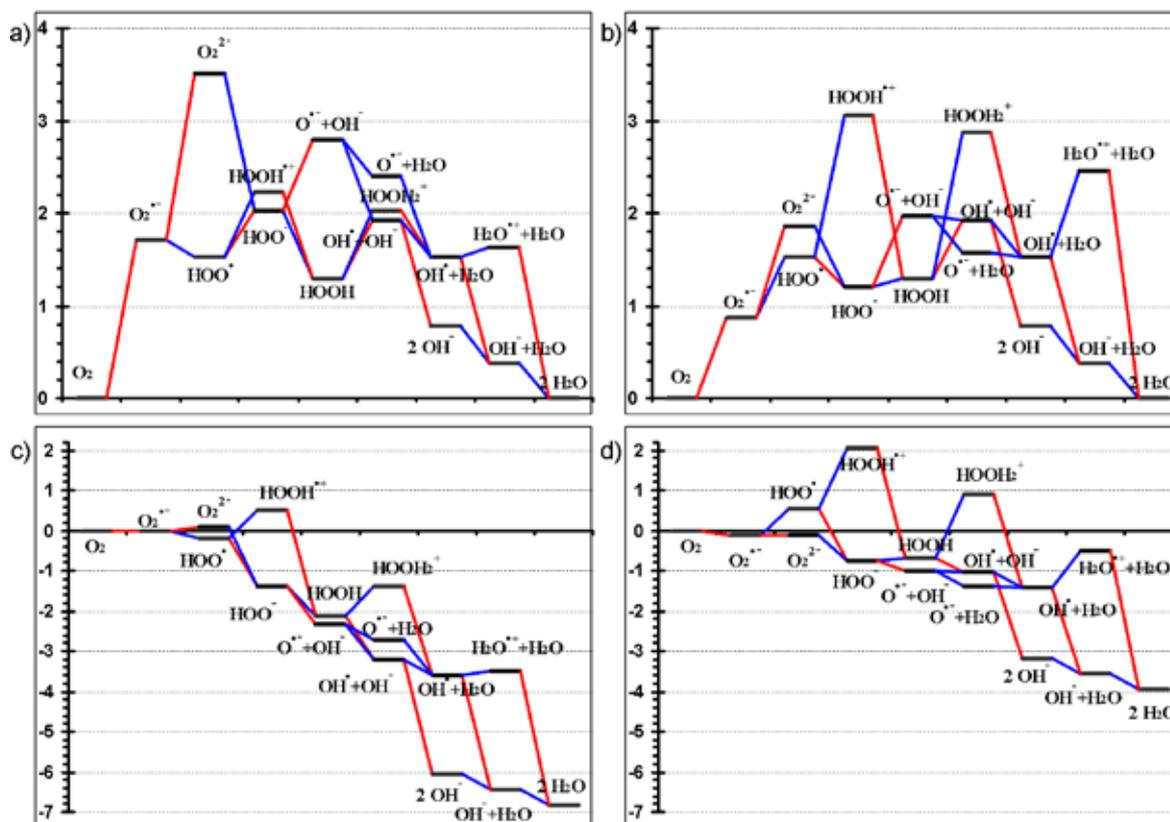
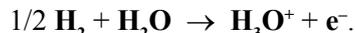


Fig. 2: Energy diagrams (eV) for the electrochemical oxygen reduction reaction at (a) pH 0, +1.28 V, (b) pH 14, +0.45 V, (c) pH 0, -0.43 V, (d) pH 14, -0.54 V.



The water autoprotolysis reaction is a demanding test for solvation models, as two charged species are generated from two neutral species. The experimental reaction free energy is 1.034 eV. In the standard hydrogen electrode, the free energy of the electron corresponds to the absolute potential of 4.62 V determined by Trasatti [6].

The implicit solvation model S2 (PCM) recovers about 3/4 of the solvation effect emphasizing the importance of the electrostatic contributions. One shell of explicit solvation (model S3) recovers only 2/3 of the solvation energy and a second shell (model S5) still less than the simple PCM model. Model S4 combining an explicit first solvation shell with implicit solvation performs best giving results close to the experimental values.

**Application to the electrochemical oxygen reduction.** The best performing solvation model, S4, based on a first solvation shell and the polarizable continuum approach, was used to study the oxygen reduction in aqueous solution. The detailed reaction mechanisms shown in Fig. 2 were derived from the relative energies of the intermediates proposed in Tab. 1 as a function of pH and electrode potential. Under all reaction conditions, the first step is a one-electron reduction to give a superoxide anion  $\text{O}_2^-$ . Oxygen does not accept a proton in aqueous solution. The hypothetical  $\text{HOO}^+$  would immediately transfer the proton back to a water molecule. Diagrams 2a and 2b correspond to open circuit potential, where oxygen is in equilibrium with the final product, two water molecules. At pH = 0 this corresponds to a calculated electrode potential of 1.28 V, which agrees

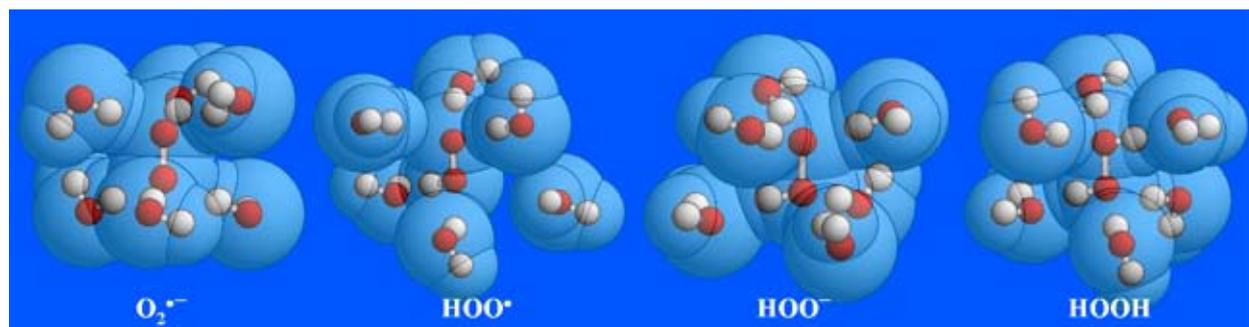


Fig. 3: Structures of selected intermediates with solvation shell.

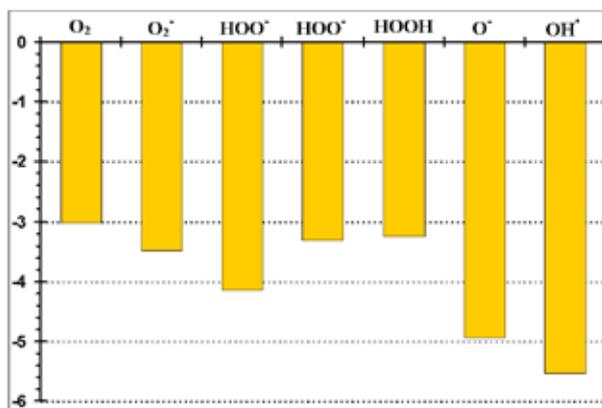


Fig. 4: Hydrogen affinities in eV as measure for the reactivity of the intermediates.

well with an experimental value of 1.23 V. At pH = 14, the calculated potential is 0.45 V. Note that the relative stability of the intermediates  $O_2^{\bullet-}$ ,  $HOO^{\bullet}$ ,  $HOO^-$ , and  $HOOH$  formed by alternating reduction and protonation steps changes dramatically with the pH, indicating different rate determining steps.

Diagrams 2c and 2d correspond to the electrode potential, where all reaction steps are exothermic or at least in equilibrium. At pH = 0 and -0.45 V this is the first electron transfer,  $O_2 + e^- \rightarrow O_2^{\bullet-}$ . At pH = 14 and -0.54 V this is the second step,  $O_2^{\bullet-} + e^- \rightarrow O_2^{2-}$  resulting in an initial two-electron reduction of oxygen. The peroxide dianion  $O_2^{2-}$  was a higher energy state not participating in the minimum energy pathway under the other reaction conditions.

The structure of selected intermediates is shown in Fig. 3.  $O_2^{\bullet-}$  and  $HOO^-$  have been detected experimentally, e.g., during oxygen reduction on gold in alkaline solution by surface sensitive spectroscopic techniques [7,8].

**Discussion and Conclusions.** The above results give a detailed mechanism for the electrochemical reduction of oxygen in aqueous solution in terms of elemental steps. They also show how the minimum energy reaction path changes with the pH and electrode potential indicating different rate determining steps. The present model allows to

predict reaction conditions that stabilize particular intermediates enhancing their formation. This allows optimization of conditions for their detection.

In the current calculations, the electrocatalytic effects of an electrode surface are not included. However, the present mechanism may be interpreted as the generic or default mechanism always available, even on a poisoned or catalytically inactive surface. Furthermore, the energies given in Fig. 2 correspond to the energy of species that have desorbed from the electrode surface and hence may diffuse away from the electrode into the reaction medium.

Several of these Intermediates are highly reactive radicals that may damage an organic coating, e.g., by hydrogen abstraction. The chemical degradation started by such a reaction may ultimately lead to de-adhesion. The reactivity in hydrogen abstraction reactions (hydrogen affinity) is illustrated in Fig. 4. The hydroxyl radical  $OH^{\bullet}$  is the most reactive intermediate, even more reactive than the superoxide and peroxide radicals  $O_2^{\bullet-}$ , and  $HOO^{\bullet}$ . Further studies will be necessary to understand the reaction cascades that may be started by these reactive species.

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## Material Point Homogenization based on a Novel Finite-Strain Grain Cluster

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Many metallic materials used for structural applications are of polycrystalline nature, i.e., they contain a very large number of grains. The plastic response of an individual grain, assuming deformation by slip of dislocations, is anisotropic due to its crystalline structure. Therefore, such anisotropy can be noticeable for polycrystals in which the crystallite orientation distribution (texture) of the material is not random. In this context it should be noted that practically all process routes lead to texture evolution. Predicting the texture evolution during forming operations of metallic materials is of high economical interest. An important example is sheet metals produced by rolling which typically possess strong textures. The resulting anisotropies influence subsequent deformation, with consequences to tool design, crash behavior or surface quality of final products like for instance beverage/food cans, automotive body parts, or casings for consumer electronics.

Modelling the interaction of lattice slip and evolving grain orientations in a polycrystalline setting started in the first half of the 20th century with the works of Taylor [1] and Sachs [2] but remains an ongoing challenge. The reason is that in a polycrystal the neighborhood around individual grains as well as their shape varies from grain to grain. Therefore, the local boundary conditions experienced by each grain differ more or less strongly from the global average boundary conditions applied to the polycrystal as a whole. Solving this boundary value problem with all degrees of freedom present in a polycrystal is certainly unfeasible when dealing with several million grains. Hence, simplifications are required to be able to model and predict the coupled deformation behavior and texture evolution at a continuum material point.

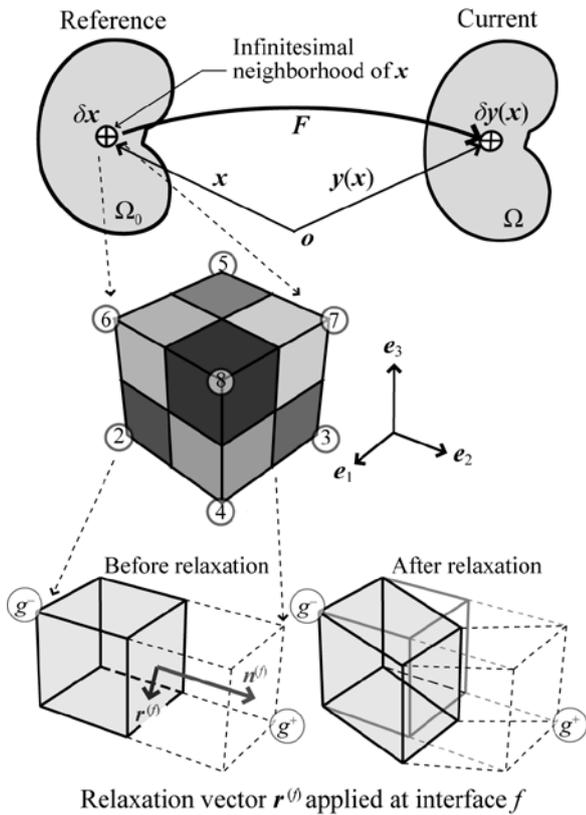
Several advancements of the Taylor model are available that lead to improved texture predictions. For example, relaxed-constraints models [3–6] diminish the strict Taylor assumption of an equal strain (rate) tensor for each grain by allowing for deviations in one or several components of each strain tensor. This may be justified for special anisotropic (e.g. flat or elongated) grain shapes but it restricts these models to specific applications and deformation modes (e.g. rolling). Beyond such global relaxations, grain interactions are not considered at all.

By contrast, grain cluster models compose a mesoscopic aggregate of interacting grains (typically two or eight) for which the boundary conditions are jointly imposed. The deformation can be relaxed among the grains within a cluster as long as the external boundary conditions are fulfilled for the entire cluster. The most simple grain cluster model is the so-called LAMEL model introduced by Van Houtte and coworkers [7,8]. It considers a stack of two grains that are free to move their shared interface in its plane. The inherent anisotropy due to a single stacking direction can be overcome by the so-called advanced LAMEL model (ALAMEL [9]), which considers grain interactions in a statistical way and divides each grain into a number of grain boundary regions treated in the spirit of LAMEL. Another route is followed by the GIA model of Wagner [10] (see [11] and references therein for further developments) which considers aggregates of  $2 \times 2 \times 2$  grains and is hence applicable to general deformation modes. A characteristic feature of the GIA model is the penalizing of incompatibilities, which develop among grains, by assigning energy contributions to them. Those are associated with the build up of geometrically necessary dislocations at the grain boundaries. The consideration of these energies renders the GIA model material-dependent but much more difficult to solve than other Taylor-type models.

We consider the macro-scale material point to also be a cluster of  $2 \times 2 \times 2 = 8$  hexahedral grains of (initially) equal shape and size which is imagined to be periodically repeated in all three dimensions (Fig. 1). In our novel setup termed “Relaxed Grain Cluster” (RGC), the finite-strain framework is adopted. The quantities of interest, i.e., the effective deformation gradient,  $\bar{\mathbf{F}}$ , and work-conjugate first Piola–Kirchhoff stress,  $\bar{\mathbf{P}}$ , of the grain cluster, then follow as volumetric average from the per grain quantities,  $\mathbf{F}^{(g)}$  and  $\mathbf{P}^{(g)}$ :

$$\bar{\mathbf{F}} = \frac{1}{8} \sum_{g=1}^8 \mathbf{F}^{(g)} \quad \text{and} \quad \bar{\mathbf{P}} = \frac{1}{8} \sum_{g=1}^8 \mathbf{P}^{(g)}$$

where spatial homogeneity within each grain is assumed. The stress per grain results from the (known) material constitutive law  $\mathbf{P}^{(g)}(\mathbf{F}^{(g)})$ .



**Fig. 1:** Admissible relaxation at one particular interface within the grain cluster considered at each continuum material point.

The kinematic relaxation of the cluster can be conveniently described by relaxation vectors,  $\mathbf{r}^{(f)}$  with  $f = 1, \dots, 12$ , each attached to one internal interface and characterizing the displacement of the attached interface (Fig. 1 bottom). By this, equal but opposite deviations of the local deformation gradient in grains  $g^-$  and  $g^+$  from the cluster average follow:

$$\Delta \mathbf{F}^{(g^-)} = +\frac{2}{1/8} \mathbf{r}^{(f)} \otimes \mathbf{n}^{(f)} \text{ and } \Delta \mathbf{F}^{(g^+)} = -\frac{2}{1/8} \mathbf{r}^{(f)} \otimes \mathbf{n}^{(f)}$$

In accord with classical relaxed-constraints models the resulting 36 possible relaxations are determined by minimizing the cluster's total energy density, which is taken as sum of the average energy densities resulting from deformation work and interfacial incompatibility. It turns out that the stationary point of the total energy density with respect to each  $\mathbf{r}^{(f)}$  is equivalent to a stress balance on the corresponding interface  $f$ :

$$\left( \mathbf{P}^{(g^+)} + \mathbf{R}^{(g^+)} - \mathbf{P}^{(g^-)} - \mathbf{R}^{(g^-)} \right) \cdot \mathbf{n}^{(f)} = \mathbf{0}$$

The stress-like mismatch penalty  $\mathbf{R}^{(g)}$  depends on the shear modulus  $\mu$ , Burgers vector length  $b$  and grain dimensions (vector)  $\mathbf{d}$  of the material in question:

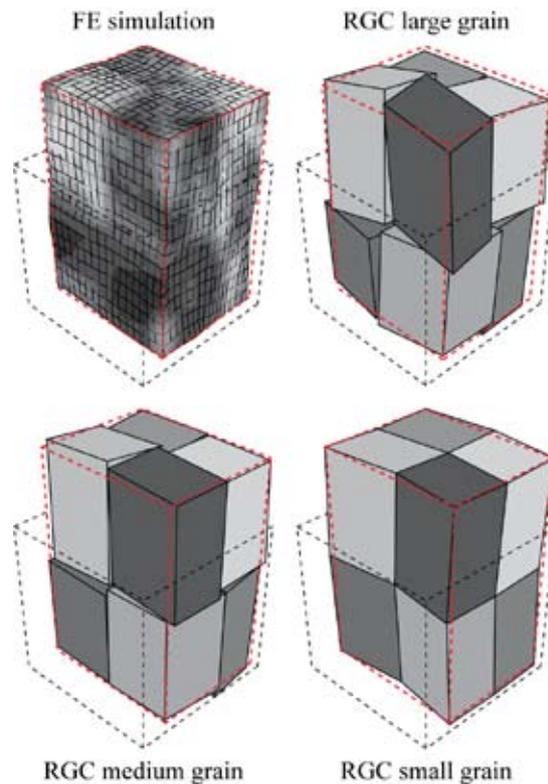
$$\left[ \mathbf{R}^{(g)} \right]_{ij} = \sum_{f \in f^{(g)}} \frac{C}{\mathbf{d} \cdot \mathbf{n}^{(f)}} \frac{\mu b}{\|\mathbf{M}^{(f)}\|} \left[ \mathbf{M}^{(f)} \right]_{is} \left[ \mathbf{n}^{(f)} \right]_r \varepsilon_{srj}$$

with  $C$  being an adjustable parameter and  $\varepsilon_{srj}$  the permutation (Levi-Civita) tensor.

The mismatch tensor  $\mathbf{M}^{(f)}$  quantifies the deformation incompatibility across each interface, which may result from neighboring relaxations, and is defined as:

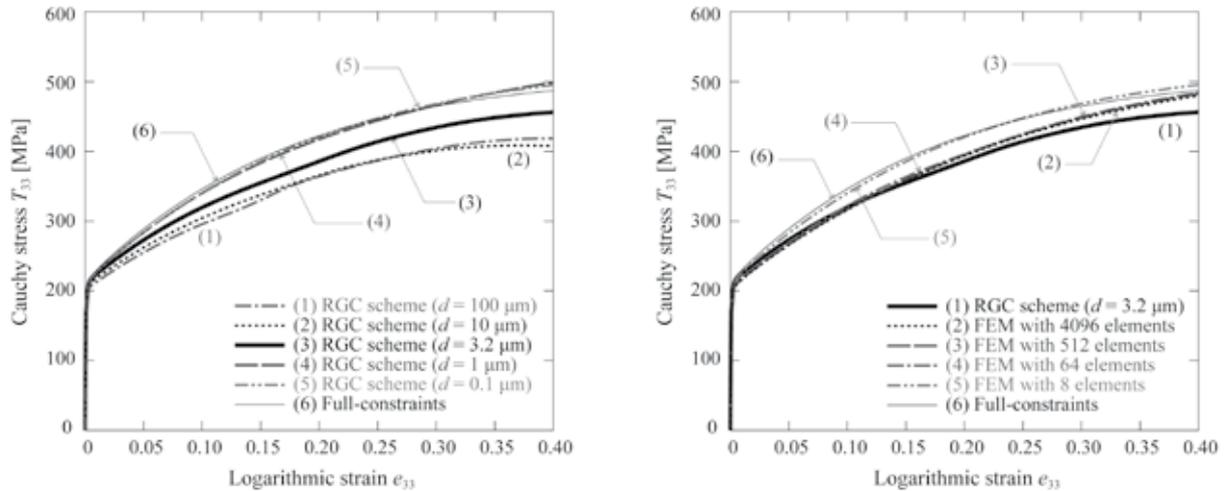
$$\left[ \mathbf{M}^{(f)} \right]_{ij} = -\left[ \mathbf{n}^{(f)} \right]_r \left( \left[ \mathbf{F}^{(g^+)} \right]_{is} - \left[ \mathbf{F}^{(g^-)} \right]_{is} \right) \varepsilon_{jrs}$$

To assess the quality of this homogenization scheme we compared the predicted mechanical behavior of one cluster – with a particular grain orientation arrangement – to direct finite element (FE) simulations of the same cluster at different mesh resolution.



**Fig. 2:** Comparison of predicted shape change under unidirectional tension for a particular grain orientation arrangement.

Fig. 2 presents the FE solution for the finest mesh together with three RGC results at different grain sizes. As expected, the interfacial mismatch observed in RGC is decreasing with decreasing grain size. The corresponding grain size-dependent stress–strain response is shown in Fig. 3 (left). A lower (upper) limiting strength is observed at large (small) grain sizes. The upper limit in strength is not as high as the corresponding iso-strain result (not shown) since still three en-bloc relaxations are possible despite virtually zero interface mismatch (Fig. 2 lower right). The highest sensitivity to grain size is observed between 1 and 10  $\mu\text{m}$ . The quantitative comparison of the stress–strain response (Fig. 3 right) as well as of the per-grain deformation (Fig. 2 left) indicates that the closest match between RGC and high-resolution direct FE is achieved at intermediate grain sizes of approx. 3  $\mu\text{m}$ . This can be correlated



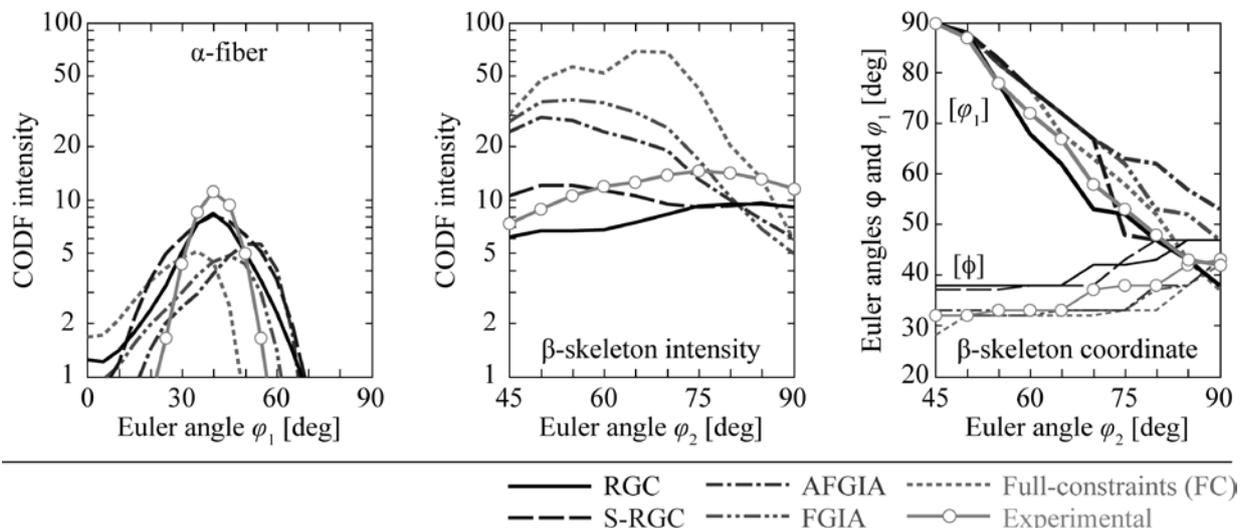
**Fig. 3:** Grain size-dependence of RGC stress response (left) and mesh-dependence of FE response (right) under unidirectional tension in direction 3.

to the relative maximum in the ratio of interfacial energy to deformation work occurring around those grain sizes and thus mimicking the FE constraint of nodal connectivity while still allowing for differences in the bulk response of each grain. The reduction of computational effort is significant: the RGC scheme is solved more than 3 (2) orders of magnitude faster than the FE simulations with 4096 (512) elements, respectively.

We additionally compared the texture evolution resulting from the RGC scheme to four other (cluster) homogenization schemes. Cold rolling of a commercial AA3104 aluminum alloy to 90% thickness reduction served as test case. The other relaxation schemes are S-RGC, which differs from RGC by the restriction  $\mathbf{r}^{(f)} \cdot \mathbf{n}^{(f)} = 0$ , thus only allowing for shear relaxation but excluding normal relaxation. The FGIA scheme follows similar concepts as the RGC but is formulated in the symmetric small-strain framework. The AFGIA differs from the FGIA by not enforcing symmetric relaxations.

Fig. 4 presents the intensity of the crystallite orientation distribution function (CODF) on the fcc  $\alpha$ -fiber and  $\beta$ -skeleton as function of  $\varphi_2$  (Euler angles  $\varphi_1$ ,  $\phi$ , and  $\varphi_2$  according to Bunge [12]). The RGC and S-RGC schemes most closely match the experimental intensity distribution. The  $\beta$ -skeleton intensity exhibits a systematic reduction between the  $\{112\}\langle 11\bar{1} \rangle$  orientation at  $\varphi_2 = 45^\circ$  (Cu component) and the  $\{123\}\langle 634 \rangle$  orientation at  $\varphi_2 \approx 63^\circ$  (S component) in the order of FC, FGIA, AFGIA, S-RGC and RGC. The intensity close to the  $\{110\}\langle 1\bar{1}2 \rangle$  orientation at  $\varphi_2 = 90^\circ$  (Brass component), however, is not influenced to a large extent by the choice of homogenization.

The development and in-grain orientation scatter of the Cu, S, and Brass components were studied in detail in some earlier works [13–16] and crucially depend on the details of the boundary conditions imposed in a model calculation. These studies showed that the development of those components is promoted by the relaxation of certain grain-to-



**Fig. 4:** Selected texture intensity after 90% thickness reduction (plane-strain) predicted by various cluster homogenization schemes in comparison to experimental reference.

grain shear constraints within a grain cluster or for the overall aggregate (in the case of a simpler model). Specifically, the exact position and intensity of the Brass component seemed to depend on the micromechanical relaxation of the shear between rolling and transverse direction (RD and TD). The current results clearly show that this orientation is much less sensitive to the details of relaxation and strain constraints than are the S and Cu orientations. This is not surprising since the Brass orientation is also the stable Sachs orientation, which is formed when using a simple Tucker stress state in conjunction with single crystal mechanics and no strain constraints. This means that from an fcc single crystal Schmid estimate the crystallographic  $\{110\}$ -direction is gradually oriented parallel to the compression axis and the  $\langle 1\bar{1}2 \rangle$ -axis parallel to the rolling direction. Since the slip systems with the highest Schmid factors also occur as dominant systems in polycrystal models which use strain-constraints one can conclude that the Brass orientation might be less sensitive to changes in the boundary conditions than the other two components.

The systematic decrease of intensity between Cu and S component coincides with an increase in the degree of relaxation allowed by the different schemes. For a cubical cluster of eight grains the maximum number of constraints possible in the framework of (symmetric) small incremental strain is 8 grains  $\times$  6 strain components = 48 while in the finite deformation framework it is 8 grains  $\times$  9 deformation components = 72. In either framework, the full-constraints Taylor homogenization allows for zero relaxations. Analyzing the remaining relaxation schemes reveals the sequence of increasing number of possible relaxations to be FGIA (14), AFGIA (20), S-RGC (24), and RGC (36). This finding is in-line with the observation that the S component is in fcc crystals generally much less stable than the Brass component. Beaudoin et al. [17] showed by detailed crystal plasticity FE simulations for a grain cluster aggregate consisting of eight different variants of the S orientation that this component tends to undergo strong in-grain orientation fragmentation. The orientation spread that was observed after plane-strain deformation of this grain ensemble extended through the entire  $\beta$ -fiber. This earlier observation underlines that the S component is very sensitive to the internal boundary conditions as is also evident from the large deviations among the different models observed in the present results.

The results for the Cu component after 90% thickness reduction show two groups with different tendency. The first class of rather stiff models (full-constraints, FGIA, and AFGIA) predicts a quite strong orientation density. The second class of more compliant models (S-RGC and RGC) predicts a more modest orientation density for this component. This result is interesting and reflects the different model

approximations and their effect on the different texture components. The Cu orientation is relatively close ( $7^\circ$ ) to the Taylor orientation  $\{4\ 4\ 11\} \langle 11\ 11\ \bar{8} \rangle$ , which is the most stable orientation in full-constraints Taylor simulations for fcc crystals. This means that the Cu orientation is promoted by stiff boundary conditions. However, if these stiff constraints are relaxed, the orientation density in the Cu component drops. This characteristic behavior of the Cu component is in contrast to the high stability of the Brass component, which does not change much under different relaxation modes. The Brass component is stress-stabilized as pointed out above, i.e., the main slip systems which are activated under plane-strain will under most relaxation states dominate the deformation of this component. The Cu orientation, in contrast, is strain-stabilized. This means that its stability does not result from a characteristic set of highly stressed slip systems such as in case of the Brass orientation. The active slip systems (under plane-strain) of the Cu orientation are not so highly stressed as in case of the Brass orientation and, therefore, depend more sensitively on the boundary conditions. This means that the closer the constraints of a model are to the full-constraints Taylor assumption, the stronger the orientation density of the Cu component should be. This is reflected by the current results.

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## Chemical Interdiffusion in Compounds of Tool Steels: Thermodynamic and Kinetic Calculations

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In tool steels produced by conventional metallurgy the evolution of the microstructure is mainly dominated by the alloy system as well as the formation of non stoichiometric mixed phases. To overcome certain restrictions of cast material, tool steels can also be processed by means of powder metallurgy (PM). A niche application of this group of steels, especially the subcategory of cold work tool steels, is their use as wear resistant materials and coatings. In recent years, several concepts have been developed, one of them being HIP-cladding [1], for producing thick wear resistant coatings on tough substrate materials. Depending on the application, the tool steel coating alone provides the required wear resistance or it is provided with additional hard particles to obtain a metal matrix composite material.

For the purpose of HIP-cladding, pre-alloyed powders are used exhibiting powder grain sizes ranging from 40  $\mu\text{m}$  to 150  $\mu\text{m}$ . These powders show a small sintering activity and are usually processed by means of hot isostatic pressing (HIP). A tool steel powder or a mixture of it with additional carbides is filled into gas-tight capsules and consolidated by HIP.

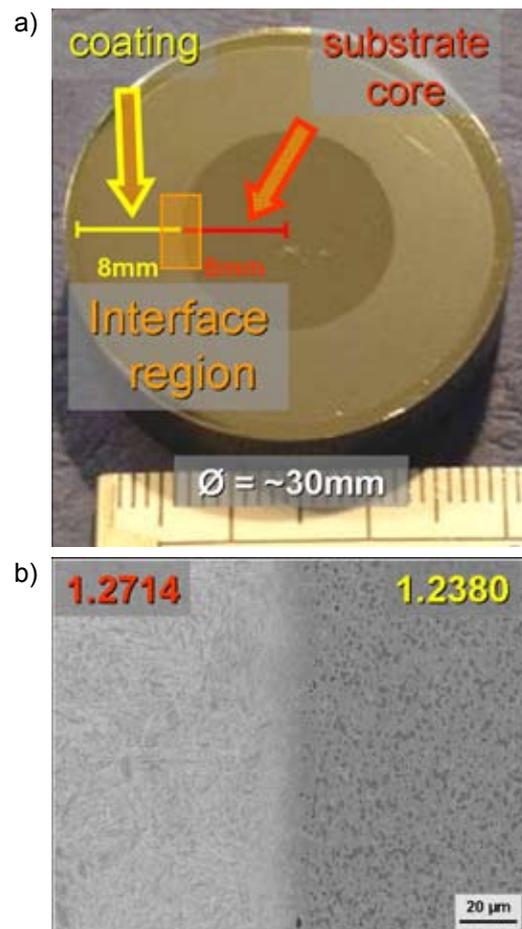
Direct hot extrusion is considered as a possible alternative for producing semi-finished parts with a large aspect ratio and the objective of a full densification by the hydrostatic pressure in the die of the extrusion press. The different steps are comparable to those of HIP processing. The powders or powder mixtures are filled into a capsule, which is evacuated, sealed, preheated in a furnace for several hours and subsequently pressed. During the preheating a sintering of the powder particles takes place, influencing the deformation behavior of the capsule in the extrusion press [2]. The basic concepts of this process and resulting materials are described in detail elsewhere [3,4].

A further development of the hot extrusion of abrasion resistant coatings was achieved by inserting a massive steel bar into the capsule and filling the retained cavity with powder. The co-extrusion of this massive and powdery materials leads to a complete densification and the formation of a tough substrate coated with a thick wear resistant layer. Depending on the materials combination and the heat treatment, differences in the formation of the interface substrate-coating can be determined. To support and explain

experimental findings, the chemical interdiffusion of one compound consisting of two different tool steels was simulated using the software Dictra (Diffusion Controlled TRANSformation, Thermo-Calc AB, Stockholm, Sweden) [5].

**Experimental.** For obtaining the compound materials, a gas-atomized cold work tool steel powder of 1.2380 was selected to serve as coating. A hot work steel bar made of 1.2714 with a diameter of 30 mm was chosen as the substrate core.

This bar was put into large capsule ( $\varnothing = 78 \text{ mm}$ ,  $l = 200 \text{ mm}$ ) made of a commercial austenitic stainless steel (1.4301) and the surrounding space filled



**Fig. 1:** Tool steel compound consisting of a substrate core of 1.2714 and coating of 1.2380 processed by hot extrusion: (a) Overview of the cross section of an extruded rod; (b) SEM micrograph of interface region depicting a fully martensitic substrate and a coating with a high volume fraction of dispersed carbides.

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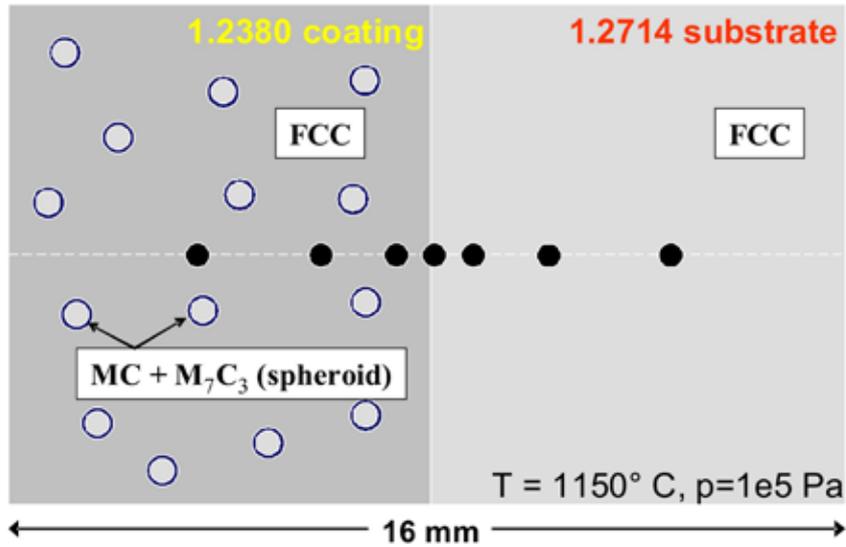


Fig. 2: Schematic layout of the 1-cell simulation with Dictra combining the homogenization model with the model for dispersed phases.

with the steel powder of 1.2380. The capsule was evacuated, sealed by TIG welding, preheated at 1150 °C for 2h and extruded with a ram speed of 36 mm/s and a pressing ratio of 5.2 : 1 into rods with a diameter of 35 mm (Fig. 1a). One sample was heat treated additionally for 8h at 1150 °C to enlarge the interface region.

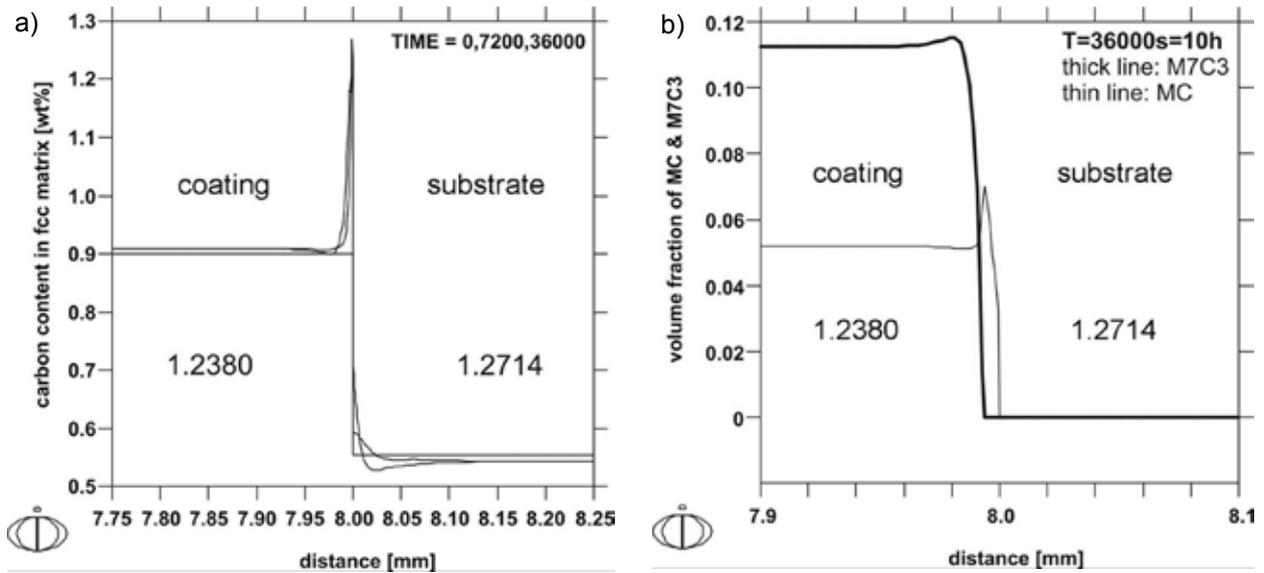
Due to the hot extrusion the steel powder is consolidated and bonded to the massive substrate material while the substrate itself is also deformed during the process. In the end, an extruded bar with an outer diameter of approximately 35 mm is formed. The resulting microstructure at the interface between substrate and coating after a typical heat treatment (30 min at 1070 °C / air + 3 x 2 h at 520 °C / air) is depicted in Fig. 1b. The micrograph shows a fully martensitic substrate, an interface region of 15-20 µm in width and a coating consisting of a martensitic matrix with a large volume fraction of dispersed carbides.

**Thermodynamic Equilibria and Diffusion Calculations.** Calculations of the thermodynamic equilibria of 1.2380 and 1.2714 were performed using Thermo-Calc with the TCFE4 database and served as the basis for the diffusion calculations. The results are given in Table 1, predicting a fully austenitic structure of 1.2714 and a 3-phase equilibrium for 1.2380 at 1150 °C. The structure of the carbides was verified experimentally by XRD and EBSD and is in agreement with the calculated data.

Combining the homogenization model and the model for dispersed systems, both included in the Dictra software package, a simulation layout as depicted in Fig. 2 was set up. The databases TCFE4 and MOB2 [6] were used for the calculation. One cell with a size of 16mm (8mm substrate + 8mm coating) was provided with an “FCC” matrix phase and initial concentration profiles as given by the equilibrium calculation (Tab. 1). Additionally, on the coating side of the cell, two dispersed phases, “M<sub>7</sub>C<sub>3</sub>” and “MC”,

Material	V <sub>f</sub>	Chemical composition [Wt.-%]								
		[%]	C	Cr	Mo	V	Mn	Si	Cu	Ni
<b>1.2380</b>										
FCC_A1#1 (Austenite)	82.6	0.9	7.63	0.77	0.67	0.39	0.65	-	-	bal.
M <sub>7</sub> C <sub>3</sub>	12.8	8.74	46.44	1.54	7.75	0.32	-	-	-	
FCC_A1#2 (MC carbide)	4.6	15.82	14.43	7.13	60.09	0.02	-	-	-	bal.
<b>1.2714</b>										
FCC_A1#1 (Austenite)	100	0.56	1.15	0.46	0.08	0.75	0.29	0.11	1.74	bal.

Tab. 1: Equilibrium phases and corresponding compositions of 1.2380 and 1.2714 at T=1150 °C and p = 101325 Pa calculated with Thermo-Calc using the TCFE4 database.



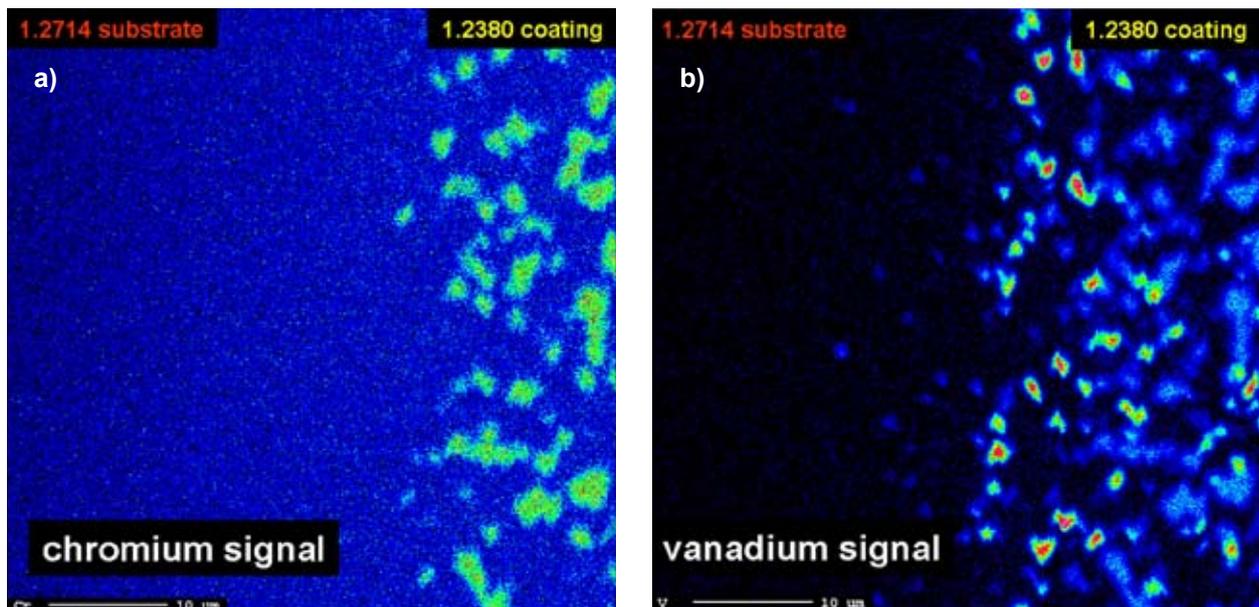
**Fig. 3:** Profiles of matrix carbon content and carbide volume fractions calculated with DICTRA: (a) Profiles of the carbon content of the FCC matrix calculated for 2 h and 10 h at 1150 °C; (b) Profiles of the carbide volume fractions of  $M_7C_3$  (thick line) and MC (thin line) calculated for 10 h at 1150 °C.

were introduced according to their equilibrium volume fractions. The isothermal calculation was supposed to cover the heat treatment time at a temperature of 1150 °C for 2 h and 10 h.

**Calculated and Experimental Results.** Element profiles measured by EMPA are in good agreement with the calculated ones. Looking at the profile of the carbon concentration in the FCC matrix, a depletion on the substrate side and an enrichment at the interface towards the coating can be recognized (Fig. 3a). These profiles are a result of carbon uphill diffusion as the activity of C in 1.2714 is higher than in 1.2380. Measurements of the microhardness across the interface exhibit a decreasing hardness on the substrate side towards the interface and a

peak in hardness at the interface. This finding is in agreement with the carbon profile calculated, considering the influence of the dissolved carbon on the hardenability. Regarding the volume fractions of carbides, a shift of the initial interface with respect to the concentration of  $M_7C_3$  carbides towards the coating is predicted (Fig. 3b). Furthermore, an increase of the volume fraction of MC carbides is calculated for the interface.

Element mappings of chromium and vanadium measured using EPMA can be related to the presence of  $M_7C_3$  and MC carbides in the matrix, as their compositions are differing significantly (cf. Tab. 1); see Fig. 4. The maps disclose a shift of chromium-rich  $M_7C_3$  carbides towards the coating side as well as an



**Fig. 4:** Element mappings of chromium (a) and vanadium (b) measured with EPMA showing a shift of the chromium-rich carbides of  $M_7C_3$  type towards the coating and an enrichment of vanadium-rich carbides of MC type at the interface.



enrichment of vanadium-rich MC carbides close to the initial interface. These results were cross-checked and verified by EBSD measurements. It can thus be stated, that the results of the Dictra calculation regarding the change in the carbide volume fractions is in full agreement with the experimental findings.

**Résumé.** Based on calculated phase equilibria of two different tool steels it was possible to define a layout for a Dictra calculation combining the models for homogenization and dispersed systems. The results of the calculation are in agreement with EPMA measurements and give an explanation for the unexpected evolution of microhardness measured across the interface. Current work deals with incorporating the influence of additional carbide particles to the coating into the calculations.

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## PART IV.

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Stratmann, Martin, Prof. Dr. (*head of department*)

Abts, Andre, 2007-04-01 to 2008-03-31, since 2008-06-01

Ankah, Genesis Ngwa, M.Sc. (Cameroon)

Biedermann, Paul Ulrich, Dr. rer. nat. (*group head temp.*)

Blumenau, Alexander Thorsten, Dr. rer. nat. (*group head*), until 2008-08-31

Borissov, Dimitar, Dr. (Bulgaria)

Borodin, Sergiy, M.Sc. (Ukraine)

Bruder, Katrin, Dipl.-Phys. until 2008-09-30 (*guest* since 2008-10-01)

Büttner, Angela, Dr. rer. nat., until 2007-12-31

Chen, Ying, M.Phys.Chem. (China)

De la Fuente, Daniel (*guest*), 2007-05-01 to 2007-12-31

Dixit, Avinash (*guest*, India), 2007-05-18 to 2007-07-31

Drensler, Stefanie, since 2007-10-15

Eberlein, Thomas Andreas Georg, Dr., until 2008-02-29

Erbe, Andreas, Dr. (*group head*), since 2007-11-01

Evers, Stefan since 2007-05-01

Fenster, Jan Christian, Dipl.-Chem.

Fink, Nicole, Dr. rer. nat.

Fujita, Naomi, Dipl.-Phys. (*guest*)

Giza, Miroslav, Dipl.-Phys. (*guest*), since 2008-01-01

Giza (former Klimow), Galina, Dipl.-Chem.

Grundmeier, Guido, Prof. Dr.-Ing.

Hamou, Faycal Riad, since 2007-09-05

Hasse, Veronika, Dr. rer. nat., since 2007-07-01

Hassel, Achim Walter, Dr. rer. nat. (*group head*)

Hümann, Sascha-Peter, 2007-03-01 to 2008-10-31

Hüning, Boris, Dr. rer. nat.

Isik-Uppenkamp, Sonnur, Dr. (Turkey)

Itani, Haybat, M.Phys. (Lebanon)

Keil, Patrick, Dr. rer. nat.

Khan, Tabrisur Rahman, M.Sc.Eng. (Bangladesh), since 2007-08-09

Klemm, Sebastian, Dipl. Wirtschaftskemiker, since 2008-04-15

Klüppel, Ingo, Dipl.-Chem. (*guest*)

Krieg, Romina, since 2008-07-01

Laaboudi, Abdellaziz, Dipl.-Chem. (Morocco)

Lill, Kirsten, Dr. rer. nat. (*guest*), until 2007-12-01

Loschen, Rebekka, Dr. rer. nat., since 2007-12-01

Mardare, Andrei Ionut, M.Sc. Dipl.-Ing. (Romania)

Mardare, Cezarina Cela, Dipl.-Ing. (Romania)

Marinkovic, Ratko, B.Sc. 2007-08-23 to 2008-09-30

Merzlikin, Sergiy, Dr. rer. nat. since 2008-03-01

Milenkovic, Srdjan, Dr.-Ing. (Serbia and Montenegro)

Mrsevic, Miroslav, 2008-05-13 to 2008-09-30

Muglali, Mutlu Iskender, since 2007-09-15

Neelakantan, Lakshman, Dr.-Ing. (India)

Oezcan, Oezlem, M.Sc. (Turkey)

Özkaya, Berkem, M.Sc. (Turkey)

Pareek, Aparna, Dr. rer. nat., since 2007-07-01

Popova, Vesselina, Dipl. Chem. (*guest*, Bulgaria), since 2007-09-01

Posner, Ralf, Dipl.-Chem.

Reithmeier, Martina, Dipl.-Phys. since 2008-10-01

Renner, Frank Uwe, Dr. rer. nat. (*group head*), since 2007-11-01

Rohwerder, Michael, Dr. rer. nat. (*group head*)



- Salgin, Bekir, M.Sc. (Turkey), since 2007-02-01
- Sanders, Daniel, Dr. rer. nat.
- Santa, Monika, Dipl.-Chem. (Italy), since 2007-02-01
- Schmitt, Markus, M.Sc., Dipl. Ing.
- Schölzel, Melanie, until 2007-09-30
- Senöz, Ceylan, M.Sc. (Turkey), 2007-10-05 (*guest* since 2008-08-01)
- Smith, Andrew, Dr. rer. nat. until 2008-07-31 (*guest* since 2008-08-01)
- Spiegel, Michael, Dr. rer. nat. (*group head*), until 2007-07-31
- Swaminathan, Srinivasan, Dr.-Ing. (India)
- Thissen, Peter, Dipl.-Chem., until 2008-06-30 (*since 2008-07-01 guest*)
- Titz, Tobias, Dipl.-Phys. until 2008-04-30
- Torres, Edmanuel, M.Sc. Phys. (Colombia)
- Tsunahiro, Shimura (*guest, Japan*), 2008-08-01 to 2008-09-30
- Valtiner, Markus, Dipl. Ing. (Austria)
- Venzlaff, Hendrik, Dipl.-Chem., since 2007-08-15
- Vlasak, René, Dipl.-Phys., until 2008-03-31 (*since 2008-04-01 guest*)
- Wang, Xuemei, Dr. (China)
- Woldemedhin, Michael Teka, M.Sc., since 2008-04-01
- Wüster, Vanja, Ma.
- Yöndem, Isil (Turkey), 2008-07-01 to 2008-08-31
- Xu, Yanhui, Dr., 2007-05-01 to 2008-04-30
- Zuo, Juan, M.Sc. (China)
- Coelho, Rodrigo Santiago, Dipl.-Ing. (Brazil)
- Corpas Garcia, Maria (Spain), since 2008-03-15
- Da Fonseca Barbatti, Carla, Dr. (Brazil) until 2008-10-31
- Dumont, Maitena, Dipl.-Ing. (France), since 2007-11-01
- Dzieciol, Krzysztof Piotr, Dipl.-Ing. (Poland)
- Garcia, Jose Luis, Dr. (*group head*, Argentina), 2007-04-01 to 2008-09-30
- Guio Sanabria, Jaime Andres (Colombia), 2007-02-01 to 2007-12-31
- Juricic, Claudia, Dipl.-Ing., until 2008-07-31
- Kostka, Aleksander, Dr. (*group head*, Poland)
- Maccio Colmenares, Maria del Rosario, Dipl.-Ing. (Venezuela), since 2007-05-01
- Mauro, Martin, Dipl.-Ing. (Argentina), since 2008-04-01
- Moscicki, Marcin, Dipl.-Ing. (Poland)
- Mujica Roncery, Lais, Dipl.-Ing. (Colombia), since 2007-09-17
- Prat Borquez, Orlando Sebastian, Dipl.-Ing. (Chile), since 2008-05-15
- Preiloswki, Martin, 2007-10-01 to 2008-09-30
- Rojas Jara, David Eduardo, Dipl.-Ing. (Chile), since 2007-09-24
- Silva, Pedro Augusto, Dipl.-Ing. (Brazil)
- Sket, Federico, Dipl.-Ing. (Argentina)
- Springer, Hauke, Dipl.-Ing., since 2008-04-01
- Weber, Sebastian, Dr. (*group head*), 2007-02-01 to 2008-09-30

### Material Diagnostics and Steel Technology

- Pyzalla, Anke Rita, Prof. Dr.-Ing. (*head of department*) until 2008-09-30
- Agudo, Leonardo, Dipl.-Ing. (Venezuela)
- Amaral Sales, Lucas, since 2008-04-16
- Borbély, András, Prof. Dr. (*group head*, Hungary), since 2007-06-01
- Brito, Pedro Paiva, Dipl.-Ing. (Brazil)
- Cardinali, Daniel (Brazil), 2007-11-01 to 2008-05-31
- Cavalcanti Pinto, Haroldo, Dr. (*group head*, Brazil)
- Cerceau Isaac, Neta Augusta, Dipl.-Ing. (Brazil)

### Materials Technology

- Frommeyer, Georg, Prof. Dr.-Ing. (*head of department*)
- Bausch, Michael
- Bobrowski, Ulrich, B.Sc.
- Brokmeier, Klaus, Dipl.-Ing.
- Buchwalter, Frank, B.Sc., since 2007-10-01
- Budak, Mustafa, B.Sc.
- Deges, Johannes, Dipl.-Phys.
- Duarte, Liliana (*guest*, Switzerland), 2007-01-15 to 2007-02-16

Eleno, Luiz Tadeu Fernandes, M.Sc. (Brazil), until 2008-02-29

Engberding, Nico, Dipl. Ing.

Gnauk, Joachim, Dr. rer. nat. (*group head*), until 2007-08-31

Hanus, Pavel, Dr. (Czech Republic), since 2008-08-01

He, Cuiyun, Dr. (China), since 2008-05-01

Hotar, Adam, Dr. (Czech Republic), since 2008-08-01

Ishikawa, Shigehiro (Japan), 2007-10-01 to 2008-03-31

Jiménez, José A. (*guest*, Spain), several times, five month altogether

Krein, Ronny Christian, Dipl. Ing., until 2008-06-30

Kumar, Sharvan, Prof. (*visiting scientist*, USA), 2007-08-01 to 2007-08-31 and 2008-06-03 to 2008-06-18

Maier, Alois, Dipl. Ing. (Austria)

Marinković, Ratko, B.Sc., until 2008-09-30

Palm, Martin, Dr. rer. nat.

Prymak, Oleg, Dr. rer. nat.

Rablbauer Ralf, Dr.-Ing. (*group head*), until 2008-10-31

Schiefer, Christian, Dipl.-Phys., since 2008-04-15

Schuster, Julius C., Prof. Dr. (*visiting scientist*, Austria), 2008-10-05 to 2008-10-10

Siggelkow, Lisa (*guest*), until 2007-11-17

Stein, Frank, Dr. rer. nat.

Voß, Simon, M.Sc., since 2007-05-01

Wenke, Rainer, Dipl.-Phys.

Wittig, James, Prof. (*visiting scientist*, USA), 2007-06-01 to 2007-06-30

Zeller, Susanne, Dipl. Ing. (Austria)

### Microstructure Physics and Metal Forming

Raabe, Dierk, Prof. Dr.-Ing. (*head of department*)

Abdelkader, Nebatti, Ph.D. (*guest*, Algeria), 2008-10-01 until 2008-11-30

Agate, Nellie, student (*guest*, UK), 2008-07-28 to 2008-09-25

Aghajani Bazazi, Ali, M.Sc. (Iran)

Ayodele, Segun Gideon, M.Sc. (Nigeria), since 2007-10-08

Balasundaram, Keerthika, M.Sc. (India)

Bastos da Silva, Alice, Dr.-Ing. (Brazil), until 2007-12-31

Bernardi, Heide, M.Sc. (*guest*, Brazil), 2007-09-02 to 2007-12-01

Bieler, Thomas Rector, Prof. (USA), 2008-07-15 to 2008-08-15

Brahme, Abhijit, Ph.D. (USA), until 2008-08-31

Calcagnotto, Marion, Dipl. Geow., since 2007-05-07

Cao, Yanpin, Pd.D. (China), 2007-04-01 to 2008-09-14

Chen, Jinju Ph.D. (*guest*, China), 2008-06-09 to 2008-06-20

Counts, Art, Ph.D. (USA), until 2008-09-19

Crimp, Martin, Prof. (USA), 2008-07-15 to 2008-08-15

Davut, Kemal, M.Sc. (Turkey), since 2008-04-01

Demir, Eralp, M.Sc. (Turkey), since 2007-06-01

Dmitrieva, Olga, Ph.D. (Russia), since 2007-07-01

Eden, Yuval M.Sc. (*guest*, Israel), 2008-09-10 to 2008-10-31

Eisenlohr, Philip, Dr.-Ing.

Elhami, Nahid, B.Sc., 2008-01-07 to 2008-12-31

Fabritius, Helge Otto, Ph.D.

Frommert, Matthias, Dr.-Ing.

Godara, Ajay, Dr.-Ing. (India), 2004-04-15 to 2007-04-14

Gross, Markus, Dipl.-Phys., since 2008-05-01

Gutierrez, Ivan, Ph.D. (Spain), since 2007-11-01

Haas, Sergej, Dipl.-Des., since 2000-09-01

Hamidi, Nima, M.Sc. (Iran), 2008-03-01 to 2008-08-31

Hantcherli, Luc, M.Sc. (France)

He, Cuiyun, Dr. (China)

Herrera Pulgarin, Clara, Ph.D. (Colombia), since 2007-03-15

Hild, Sabine, PD Dr. rer. nat. (*group head*), 2008-05-01 to 2008-09-30

Huang, Zhiheng, Ph.D. (China), 2007-08-01 to 2008-06-15

Khorashadizadeh, Anahita, M.Sc. (Iran), since 2007-07-01

Knoll, Helene, B.Sc.

Krüger, Timm, Dipl.-Phys., since 2008-05-01



- Li, Hongyan, Ph.D. (China), 2007-06-01 to 2008-05-31
- Liu, Bing, B.Sc. (China), since 2008-07-15
- Liu, Tao, M.Sc. (China)
- Ma, Duangchen, M.Sc. (China)
- Mandal, Suvendu, M.Sc. (India), since 2008-06-15
- Mason, Darren, Prof. (USA), 2008-07-15 to 2008-08-15
- Millán, Julio, M.Sc. (*guest*, Venezuela), since 2008-04-07
- Nikolov, Svetoslav, Ph.D. (Bulgaria)
- Nnamchi, Paul, M.Sc. (Nigeria), 2007-02-01 to 2008-01-31
- Peranio, Nicola, Dr. rer. nat., since 2007-10-01
- Ponge, Dirk, Dr.-Ing. (*group head*)
- Prinz, Günter, Ph.D., 2008-05-15 to 2008-06-30
- Ratanaphan, Sutatch, M.Sc. (Thailand), 2008-04-01 to 2008-09-30
- Rasp, Wolfgang, Dr.-Ing. (*group head*)
- Raue, Lars, Ph.D., until 2008-07-31
- Ro, Yunjo, Ph.D. (Korea), since 2008-07-01
- Romano Triguero, Patricia, Ph.D. (Spain), until 2008-06-30
- Roters, Franz, Dr. rer. nat. (*group head*)
- Ruhl, Dominik, student (*guest*), 2008-07-07 to 2008-09-12
- Sachs, Christoph, Ph.D., until 2008-08-31 (*guest* since 2008-09-01)
- Sander, Benedikt, B.Sc.
- Sandim, Hugo, Prof. University of Lorena (*guest*, Brazil), 2008-02-01 to 2008-03-01
- Sandim, Maria, Ph.D., University of Lorena (*guest*, Brazil), 2008-02-01 to 2008-03-01
- Sato, Hisashi, Ph.D. (*guest*, Japan), 2008-06
- Schestakow, Igor, B.Sc., since 2007-03-15
- Schulz, Stefan, Dipl.-Ing., until 2008-11-30
- Song, Jingbo, M.Sc. (China), since 2007-10-01
- Steinmetz, David, M.Sc. (USA)
- Svirina, Julia, Ph.D. (*guest*, Russia), 2008-10-13 to 2008-12-13
- Swadener, John Gregory, Ph.D. (USA), 2008-09-15 to 2008-12-31
- Takata, Naok, Ph.D. (*guest*, Japan), 2007-03-01 to 2007-03-31
- Thiessen, Richard, Ph.D. (Canada), 2006-11-02 to 2007-06-30
- Tikhovskiy, Ilya, Dr.-Ing. (Russia), until 2007-07-31
- Tjahjanto, Denny, Ph.D. (Indonesia), since 2008-01-01
- Varnik, Fatholla, PD Dr. (Iran), until 2008-09-30
- Verbeken, Kim, Ph.D. (*guest*, Belgium), since 2007-11-01
- Wang, Leyun, M.Sc. (USA), 2008-07-15 to 2008-08-15
- Weber, Florian, M.Sc., 2007-12-01 to 2008-09-30
- Winning, Mirjam, PD Dr. (*guest*)
- Wu, Bin, M.Sc. (China), 2007-04-10 to 2008-05-15
- Wu, Guilin, Ph.D. (China), since 2007-08-17
- Wu, Xia, B.Sc. (China), since 2007-06-06
- Yang, Yiyi, M.Sc. (USA), 2008-07-15 to 2008-08-15
- Yazdipour, Nima, M.Sc. (*guest*, Iran), 2008-04-15 to 2008-06-30
- Yuan, Lei, M.Sc. (China), since 2007-05-01
- Zaafarani, Nader, Ph.D. (Egypt), until 2008-02-29 (*guest* since 2008-03-01)
- Zaefferer, Stefan, Dr.-Ing. (*group head*)
- Zambaldi, Claudio, Dipl.-Ing.
- Zobrist, Cédric, student (France), 2007-03-05 until 2007-08-31



# Scientific Honours

## 2007

*Ph.D. A. Brahme* received the Annual Student Award from Acta Materialia for his primary contribution to the manuscript '3D reconstruction of microstructure in a commercial purity aluminum', MS&T Conference, 2007-09, Detroit, USA, in Scripta Materialia 55, 75-80.

*Dr.-Ing. U. Brück, A. Bobrowski and Prof. Dr.-Ing. G. Frommeyer* received the 3<sup>rd</sup> best poster award for their poster 'Gefüge und Eigenschaften höchstfester TRIPLEX-Leichtbaustähle', 41. Metallographie-Tagung in Bremen, 2007.

*Dipl.-Ing. R. S. Coelho* received the best poster award for the poster 'Microstructure and residual Stresses in an AA6040-T4/AZ31B Friction-Stir Weld', MecaSens IV Vienna, 2007-09

*Dr.-Ing. P. Eisenlohr* has been selected within the graduate college AICES to be admitted to the circle of 'Young Researcher', 2007-07.

*H. Faul* was lent the honorary needle in silver with thanks and recognition for his 10 years honorary engagement in professional training by Industrie- und Handelskammer, Region Stuttgart, 2007-09.

*Dr.-Ing. M. Frommert, Dr. D. Dorner, Dr. L. Lahn, Prof. Dr.-Ing. D. Raabe, Dr.-Ing. S. Zaefferer*, received the best poster award for their contribution '3D Investigation of Early Stages of Recrystallization in Deformed Goss-Oriented Fe<sub>3</sub>Si Single Crystals' at the 'Third International conference on Recrystallization and Grain Growth', ReX & GG III, Jeju Island, Korea, 2007-06.

*Dr. rer. nat. T. Hickel* became a member of the selection committee of the Friedrich-Naumann-Stiftung für die Freiheit, 2007.

*Dr. rer. nat. T. Hickel* became a member of the selection committee of the Studienstiftung des deutschen Volkes e.V., 2007.

*Prof. Dr. rer. nat. J. Neugebauer* was awarded the honorar degree 'Honorarprofessor der Ruhr-Universität Bochum', 2007.

*Prof. Dr.-Ing. D. Raabe* has been elected Member of the Board of Governors of Acta Materialia Inc., USA, 2007.

*Prof. Dr.-Ing. D. Raabe* has been elected as a member into the Selection Committee of the Alexander von Humboldt Foundation, 2007.

*Ph.D. P. Romano* has been awarded a grant research residency from the Japan Society for Promotion of Science (JSPC) to the 'National Institute for Materials Science (NIMS)', 2007-08.

*Ph.D. C. Sachs* has been awarded the 'Minerva Short-Term Research Grant', from the Minerva Stiftung 2007-08.

*Ph.D. C. Sachs* has been awarded Champion H. Mathewson 2007 by TMS for the paper 'Homogeneous Steel Infiltration', 2007.

*B. Stieger* has been honoured by Max-Planck-Gesellschaft zur Förderung der Wissenschaften e.V. for his achieved performances during his education and training as 'outstanding apprentice of the Max-Planck-Gesellschaft' in the qualified job 'Mathematical-Technical-Assistent', 2007-08.

*Ph.D. K. Verbeken, N. Van Caenegem and Prof. Dr.-Ing. D. Raabe* received the best poster award for their contribution 'Identification of  $\epsilon$  martensite in Fe-based shape memory alloys by means of EBSD' at the symposium 'Microscopy and spectroscopy techniques in advanced materials characterization' on the E-MRS 2007 conference, Warsaw, Poland, 2007-09.

*Dr. rer. nat. K. Wapner* received the Otto-Hahn-Medal 2006, Kiel, 2007-06.

*Dipl.-Ing. C. Zambaldi, Dr. rer. nat. F. Roters, Prof. Dr. T. R. Bieler and Prof. Dr.-Ing. D. Raabe* were awarded from 'Chair of the 11<sup>th</sup> World Conference on Titanium (JIMIC5)' 'Certificate of merit for best poster presentation to Micromechanical Modeling of Near-Gamma Titanium Aluminides', Kyoto, Japan, 2007-06.

*Dipl.-Ing. C. Zambaldi* has been awarded a grant research fellowship from the Japan Society for Promotion of Science (JSPC) to the 'National Institute for Materials Science (NIMS)' 2007-10 to 2007-12.



## 2008

*A. Bobrowski, Dipl.-Ing. K. Brokmeier, Prof. Dr.-Ing. G. Frommeyer, Dipl.-Ing. A. Maier and Dr.-Ing. R. Rablbauer* received the 3<sup>rd</sup> best poster award for their poster 'Metallphysikalische Verformungsmechanismen hoch manganhaltiger austenitischer Stähle', 42. Metallographie-Tagung in Jena, 2008.

*Dipl.-Ing. K. Brokmeier and Prof. Dr.-Ing. G. Frommeyer* received the 2<sup>nd</sup> best poster award for their poster 'High carbon light-weight Fe-Mn TRIP/TWIP steels with improved formability and strength' at the 17<sup>th</sup> International Federation for Heat Treatment and Surface Engineering (IFHTSE) Congress in Kobe, Japan, 2008-10.

*M.Sc. V. Bubnik* received the Dean's Award of the Faculty of Information Technology, Brno University, Brno, Czech Republic, 2008.

*Dipl.-Geow. M. Calcagnotto* has been awarded a grant research residency from the Japan Society for Promotion of Science (JSPC) to the 'National Institute for Materials Science (NIMS)', 2008-09.

*Prof. Dr.-Ing. G. Frommeyer, Dr.-Ing. R. Rablbauer, Dr.-Ing. U. Brück, Dipl.-Ing. K. Brokmeier, Dr. O. Grässel* received the VDI award for innovative materials application 2008 for their work on 'Supraduktile und hochfeste Leichtbaustähle für den zukünftigen Fahrzeugbau', 2008.

*T. Gronau* was awarded for his very good result in his final exam of the Chamber of Industry and Commerce (IHK) in the qualified job 'Materials Testing with focus on Metal Engineering', 2008-01.

*T. Gronau* has been honored by Max-Planck-Gesellschaft zur Förderung der Wissenschaften e.V. for his achieved performances during his education and training as 'outstanding apprentice of the Max-Planck-Gesellschaft' in the qualified job 'Materials Testing with focus on Metal Engineering', 2008 -07.

*Dr. rer. nat. T. Hickel* was appointed head of the Advanced Study Group 'Modelling' at ICAMS Bochum, 2008.

*M.Sc. Dipl.-Ing. A. I. Mardare, Prof. Dr. A. Ludwig, Dr. A. Savan, Prof. Dr. A. D. Wieck and Dr. rer. nat. A. W. Hassel* received the best poster award for their poster 'Combinatorial microelectrochemistry with a scanning droplet cell on binary and ternary Ti, Ta and Hf alloys', International Symposium on Anodizing Science and Technology, Rusutsu, Japan, 2008-07.

*Prof. Dr. rer. nat. J. Neugebauer* was appointed Director of the Advanced Study Group 'Modelling' at ICAMS Bochum, 2008.

*Prof. Dr.-Ing. A. R. Pyszalla* was appointed 'Korrespondierendes Mitglied im Ausland der Österreichischen Akademie der Wissenschaften', 2008-04.

*Prof. Dr.-Ing. A. R. Pyszalla* became member of the German academy Acatech, 2008-04.

*Prof. Dr.-Ing. D. Raabe* was elected as a member into the class for 'Engineering and Economics' of the Nordrhein Westfälische Akademie der Wissenschaften, 2008-04.

*Prof. Dr.-Ing. D. Raabe* received the Lee Hsun Lecture Award of the Institute of Metal Research of the Chinese Academy of Sciences, Shenyang, in recognition of outstanding accomplishments by worldwide scientists in the field of materials science and engineering, Shenyang, China, 2008.

*Ph.D. L. Raue* received the Outstanding Student Researcher Award at 15<sup>th</sup> International Conference on Textures of Materials (ICOTOM 15), Carnegie Mellon University Center, Pittsburgh, USA, 2008.

*Ph.D. P. Romano* was elected as a member of the Board of Review of Metallurgical and Materials Transactions, 2008-01.

*Dr. rer. nat. M. Rohwerder* became head of the Christian Doppler Laboratory for 'Diffusion and Segregation during Production of High Strength Steel Sheet', MPIE, Düsseldorf, 2008-03.

*Prof. Dr. rer. nat. M. Stratmann* received the H. H. Uhlig Award of the Electrochemical Society, Honolulu, USA, 2008-10.

*Prof. Dr. rer. nat. M. Stratmann* has been nominated in the SAB of the Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Vienna, Austria, 2008-04.

*Prof. Dr. rer. nat. M. Stratmann* has been elected to the Senate and to the Supervisory Board of the Max Planck Society, 2008-07.



## - SCIENTIFIC HONOURS -

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*Prof. Dr. rer. nat. M. Stratmann* has been nominated Vice President of the Max Planck Society for the CPT Section, Munich, 2008-07.

*PD Dr. F. Varnik* received the best talk award (3<sup>rd</sup> Prize) at Nano- and Microfluidics Summer Meeting, Bad Honnef, 2008-08.

*Dr.-Ing. M. M. Walczak* was honoured with the Otto-Hahn-Medal 2007, Dresden, 2008-06.

*Dipl.-Ing. C. Zambaldi* was awarded with the 1<sup>st</sup> prize in the poster competition of the RMS Electron Backscatter Diffraction Meeting, Sheffield, UK, 2008.



# Participation in Research Programmes

## National:

### Alexander von Humboldt Foundation

Low-complexity algorithms and multiscale methods for large metallic systems

### BMBF

Advanced production methods combined with high sustainability of the coating of steel strips

Characterisation of final parts based on integral materials simulation along the process chain

Development of high performance materials for high-temperature heat exchanger and car-exhaust systems

High resolution EPR spectroscopy of thin silicon films for solar energy science

Modeling of multi-pass hot forging of AFP steels

New combined sustainable process technologies for surface finished steel sheets

Ultra-high strength carbon-aluminium steels (BMBF/BMWA)

Virtual development of ceramics and composite materials with tailored transport properties

### DFG

Aachen Institute for Advanced Studies in Computational Engineering Science (AICES)

Ab initio description of temperature dependent effects in dimensionally constrained magnetic shape memory Heusler alloys

Accurate Calculation of the electronic structure at grain boundaries via density functional theory and quasi particle corrections

Al-rich AlTi-alloys: Transformation of h-Al<sub>2</sub>Ti and Al<sub>5</sub>Ti<sub>3</sub> and formation of lamellar  $\gamma$ -TiAl + r-Al<sub>2</sub>Ti microstructures

Antireflecting interlayers to increase transmission through thin metal films in spectroelectrochemical experiments

Cold rolling and static recrystallisation of rare earth Mg alloys

Collaborative research centre 761: 'Steel - ab initio. Quantum-mechanically guided design of new Fe-based alloys', partial project: Ab initio derivation of Gibbs enthalpies, stacking fault energies and boundary energies at finite temperatures'

Collaborative research centre 761: 'Steel - ab initio. Quantum-mechanically guided design of new Fe-based alloys', partial project: 'Defects and residual stresses in Fe-Mn-C steels'

Elucidation of corrosion phenomena with high lateral resolution using scanning probe techniques

Experimental and theoretical investigations of the dynamics of collective phenomena in blood I: Idealized vesicle/fluid droplet models

Growth and simulations of quaternary GaInAsN quantum dots

High resolution scanning electron back scatter diffraction experiments of local crystallographic orientation patterning during plastic deformation

Interaction between grain boundaries with mechanical stresses

Interference and constraints of the impurity level of titanium alloys for recycling purposes

Investigation of size effects with respect to texture and anisotropy



Local mechanical properties of Mn-based steels

Materials world network: Physically based approach for predicting and minimizing damage nucleation in metals

Mechanical properties and interfaces of ultrafine grained materials by using 3D microscopy

Microstructural investigations of the kinetics of strain accumulation and damage development during creep of short fiber reinforced aluminum alloys

Microstructure mechanics and fundamentals of twinning

Modeling of phase and microstructure formation under non-equilibrium conditions of Ti-Al laser welded seams

Multiscale growth and doping simulations of nanostructured devices

Nanofluid mechanics

Non-equilibrium flow at gradient surfaces: Separation phenomena of multi-component fluids.

Non-equilibrium flow at gradient surfaces: Droplet kinetics and particle motion

Novel Procopiu effect based magnetic field sensors, using the INROLISP process for the production of the sensor cores.

Relationship between microstructure and damage mechanisms in multiphase steels

Research priority program SPP 1204: Algorithms for the fast materials-oriented simulation of process chains in forming technology

Research priority program SPP 1165: Nanowires and nanotubes - from controlled synthesis to function, partial project: Functional devices from directionally grown nanowire arrays

Research priority program SPP 1165: Nanowires and nanotubes - from controlled synthesis to function, partial project: Production of nanowire arrays through directional solidification and their application

Scale-bridging studies of the elastic contributions to nucleation and initial microstructure formation in the eutectic system Ti-Fe

Simulation study on scaling effects in nano- and microscale fluid dynamics at deformable metal surfaces

Synthesis, structural characterisation and investigation on mechanical properties of ultra fine-grained or nanocrystalline ordered  $\text{Fe}_3\text{Al}$  -(X=Cr,Ti) alloys.

The hierarchical structure of sauropodomorph bones as a key to exceptional body size: Insights from materials science.

### **Helmholtz Gemeinschaft**

Improving performance and productivity of integral structures through fundamental understanding of metallurgical reactions in metallic joints (IPSUS)

Photon and neutron research on advanced engineering materials (PNAM)

Theoretical and experimental multiscale modelling of Al and  $\text{AlCu}_4$

### **Max Planck Society**

$\text{Ag/TiO}_2$  nanocomposite ultra-thin films

Computational mechanics of polycrystals

Fundamental investigation of the de-alloying of CuAu and CuPd model alloys

Investigation of the fundamental mechanisms of passivity breakdown for binary alloys with main focus on STM and TEM investigations

Optimizing the loading procedure for co-deposition of silica into zinc



Oxygen reduction at self-assembled monolayer / gold interfaces  
Prediction of structures and phase transitions in solid state chemistry  
The nature of Laves phases

### **State of North Rhine-Westphalia**

International Max Planck Research School (IMPRS) for Surface and Interface Engineering in Advanced Materials (SurMat)

### **International:**

#### **Austrian Science Fund (FWF)**

Texture, strain and stress development in iron oxide scales

#### **Christian Doppler Society**

Adhesion, electronic structure and mechanics of thin amorphous films on reactive metals  
Advanced characterisation techniques for novel zinc-based alloy coating  
Analysis of the growth of thin amorphous films on zinc coated steel  
Diffusion and segregation mechanisms during production of high strength steel sheet, Module I: Selective enrichment at hot and cold rolled strip  
Diffusion and segregation mechanisms during production of high strength steel sheet, Module II: Pickling Module  
Diffusion and segregation mechanisms during production of high strength steel sheet, Module III: Hydrogen Module  
Polymer/Metall Interfaces, Module I: Understanding of the electronic and adhesive properties of passive Films on Zn alloy coatings  
Polymer/Metall Interfaces, Module II: Correlation of microstructure of the substrate and the layer formation as well as the formability of thin hybrid conversion coatings  
Polymer/Metall Interfaces, Module III: Adhesion of polymer coatings on modified Zn alloy surfaces in environments of high water activity  
Predictability of barrier and mechanical properties of organic/inorganic nanocomposite and multilayer films  
Water at polymer/metal interfaces

#### **European Science Foundation**

Ab initio description of iron and steel: Magnetism and phase diagrams (ADIS 2008)

#### **European Union**

Intermetallic materials processing in relation to earth and space solidification  
Numerical modelling / lifetime prediction of delamination polymer coating disbonding and material degradation



Nextgenbiowaste 'Innovative demonstrations for the next generation of biomass and waste combustion plants for energy recovery and renewable electricity production'

PARSEM: Interfacial phenomena at atomic resolution and multiscale properties of novel III-V semiconductors

### **RFCS**

Characterisation of thin films on rough steel substrates

Characterisation of ultra-thin films on heterogeneous substrates

Development and evaluation of coatings and surface conditions on steel for antibacterial and easy to clean properties

Formability and self repair properties of advanced weldable primers

Forming behaviour of corrosion protection primers

Induction assisted welding technologies in steel utilisation

Materials for increased performance in sustainable fuel combustion

Methodology of hydrogen measurement in coated steels

New approaches in electrolytic cleaning of cold rolled steel sheets

Novel annealing procedures for improving HDG of HSS

Self-healing at cut-edge of coil coated galvanized steel sheet

Simulation of barrier properties and formability of multilayer organic coatings

Space efficient curing methods and simulation-aided coating engineering for extending lifetime of aesthetic coil coatings

Steady reactivity in hot-dip coating by direct deposit of iron oxides

Ultra-high strength and ductile FeMnAlC light-weight steels



# Collaboration with Research Institutes and Industrial Partners

## Research Institutes

### National:

Access e.V., Aachen

Arbeitsgemeinschaft Wärmebehandlung und Werkstofftechnik e.V (AWT), Bremen

Bundesanstalt für Geowissenschaften und Rohstoffe, Hannover

Christian-Albrechts-Universität zu Kiel, Kiel

Clausthaler Umwelttechnik - Institut GmbH, Clausthal

CPM Gesellschaft für Computeranwendung, Prozess- und Materialtechnik GmbH, Herzogenrath

Deutsche Gesellschaft für Materialkunde e.V. (DGM), Frankfurt (Main)

Deutsches Elektronen-Synchrotron DESY, Hamburg

Deutsches Zentrum für Luft- und Raumfahrt e.V., Cologne

DOC Dortmunder Oberflächenzentrum GmbH, Dortmund

Forschungszentrum Jülich GmbH, Jülich

Forschungszentrum Karlsruhe GmbH, Karlsruhe

Fraunhofer-Gesellschaft zur Förderung der angewandten Forschung e.V, Munich

Fraunhofer Institut für Werkstoffmechanik, Freiburg

Fraunhofer-Institut für Werkzeugmaschinen und Umformtechnik, Dresden

Freie Universität Berlin, Berlin

Fritz-Haber-Institut der MPG, Berlin

Georg-August-Universität Göttingen, Göttingen

Gesellschaft zur Förderung der analytischen Wissenschaften e.V. ISAS, Dortmund

Gesellschaft zur Förderung der Spektrochemie und Angewandten Spektroskopie e.V., Dortmund

GKSS-Forschungszentrum Geesthacht GmbH, Geesthacht

Gottfried Wilhelm Leibniz Universität Hannover, Hannover

Hahn-Meitner-Institut Berlin GmbH, Berlin

Hamburger Synchrotronstrahlungslabor HASYLAB, Hamburg

Heinrich-Heine-Universität Düsseldorf, Düsseldorf

Helmholtz-Zentrum Berlin für Materialien und Energie, Berlin

Hochschule Karlsruhe - Technik und Wirtschaft, Karlsruhe

Humboldt-Universität zu Berlin, Berlin

Innovationsgesellschaft für fortgeschrittene Produktionssysteme in der Fahrzeugindustrie mbH, Berlin

Institut für Kristallzüchtung, Berlin

Johannes-Gutenberg-Universität Mainz, Mainz

Laser Zentrum Hannover e.V., Hannover



Leibniz Institut für Festkörper- und Werkstoffforschung Dresden e.V., Dresden  
Leibniz Institut für Polymerforschung, Dresden  
Max-Planck-Institut für chemische Physik fester Stoffe, Dresden  
Max-Planck-Institut für Festkörperforschung, Stuttgart  
Max-Planck-Institut für Kohlenforschung, Mülheim an der Ruhr  
Max-Planck-Institut für Mathematik in den Naturwissenschaften, Leipzig  
Max-Planck-Institut für Metallforschung, Stuttgart  
Max-Planck-Institut für Mikrostrukturphysik, Halle  
Max-Planck-Institut für Polymerforschung, Mainz  
Otto-von-Guericke Universität Magdeburg, Magdeburg  
Paul-Drude-Institut, Berlin  
Rheinische Friedrich-Wilhelms-Universität Bonn, Bonn  
Rheinisch-Westfälische Technische Hochschule (RWTH) Aachen, Aachen  
Ruhr-Universität Bochum, Bochum  
Technische Universität Berlin, Berlin  
Technische Universität Braunschweig, Braunschweig  
Technische Universität Clausthal, Clausthal  
Technische Universität Dresden, Dresden  
Technische Universität Munich, Munich  
Universität Augsburg  
Universität Bremen, Bremen  
Universität des Saarlandes, Saarbrücken  
Universität Dortmund, Dortmund  
Universität Erlangen, Erlangen  
Universität Hannover, Hannover  
Universität Karlsruhe (TH), Karlsruhe  
Universität Konstanz, Konstanz  
Universität Paderborn, Paderborn  
Universität Stuttgart, Stuttgart  
Universität Ulm, Ulm  
Universitätsklinikum Münster, Münster  
Universität Duisburg-Essen, Duisburg  
VDEh-Betriebsforschungsinstitut GmbH, Düsseldorf



## International:

Academy of Sciences of the Czech Republic, Brno, Czech Republic  
Akademia Gorniczo-Hunicza, Krakow, Poland  
Albion College, Albion, USA  
Aristotie University of Thessaloniki, Thessaloniki, Greek  
British Ceramic Research Ltd., Penkhull, UK  
Brown University, Providence, USA  
Carnegie Mellon University Center, Pittsburgh, USA  
Centre de Mise en Formes des Matériaux (CEMEF), Sophia Antipolis, France  
Centre De Recherches Metallurgiques CRM, Brussels, Belgium  
Centre National de la Recherche Scientifique, Paris, France  
Centre National de la Recherche Scientifique, Delegation Cote D'Azur, Valbonne, France  
Centro Nacional de Investigaciones Metalurgicas, Madrid, Spain  
Centro Sviluppo Materiali S.p.A, Rom, Italy  
CIRIMAT, Toulouse, France  
Cranfield University, Cranfield, UK  
Danmarks Tekniske Universitet Kongens Lyngby, Lyngby, Denmark  
Die Eidgenössische Technische Hochschule Zurich, Zurich, Switzerland  
Ecole Nationale Supérieure d'Ingenieurs de Caen et Centre de Recherche, Caen, France  
Ecole Polytechnique Federale de Lausanne, Lausanne, Switzerland  
European Space Agency ESA/ESTEC, Noordwijk, The Netherlands  
European Synchrotron Radiation Facility (ESRF), Grenoble, France  
Foundation for Research and Technology – Hellas, Heraklion, Greek  
Fudan University, Shanghai, China  
Fundacion INASMET, San Sebastian, Spain  
Fundacion ITMA, Llanera, Spain  
Helsinki University of Technology, Espoo, Finland  
Institut Charles Sadron, Strasbourg, France  
Institut National des Sciences Appliquées de Lyon, Lyon, France  
Institut National Polytechnique de Lorraine, Lorraine, France  
Institut National Polytechnique de Toulouse, Toulouse, France  
Institute of Chemical Problems for Microelectronics, Moscow, Russia  
Institute of Physics of Materials (IPM), Prague, Czech Republic  
Institute of Structural Macrokinetics and Materials Science RAS, Chernogolovka, Russia  
Instituto de Soldadura e Qualidade, Oeiras, Portugal  
Instituto Superior Tecnico, Lisbon, Portugal  
Intrinsic Materials Ltd, London, UK  
Joint Research Centre, Petten, The Netherlands



Katholieke Universiteit Leuven, Leuven, Belgium  
Korrosions- och Metallforskningsinstitutet AB Sweden, Stockholm, Sweden  
Kungl Tekniska Högskolan, Stockholm, Sweden  
Laboratoire des Colloïdes, Verres et Nanomatériaux, Montpellier, France  
Loughborough University, Loughborough, UK  
Magyar Tudományos Akadémia Kémiai Kutatóközpont, Budapest, Hungary  
Masaryk University, Brno, Czech Republic  
Michigan State Univ., East Lansing, USA  
National University of Ireland, Dublin, Ireland  
Nederlandse Organisatie voor Toegepast Natuurwetenschappelijk Onderzoek, Apeldoorn, The Netherlands  
Northwestern Technical University, Xi'an, China  
Oak Ridge National Laboratory (ORNL), Oak Ridge, USA  
Onderzoekscentrum voor aanwending van staal N.V. (OCAS), Zelzate, Belgium  
Oxford University Department of Materials, London, UK  
Palo Alto Research Center California, Palo Alto, USA  
Rautaruukki Oyj, Helsinki, Finland  
Recherche Et Développement Du Groupe Cockerill Sambre SCRL, Liege, Belgium  
Research Institute for Solid State Physics and Optics, Budapest, Hungary  
SINTEF Energiforskning AS, Trondheim, Norway  
Slovak Academy of Science, Bratislava, Slovakia  
Stanford University, Palo Alto, USA  
Swedish Institute for Metals Research, Stockholm, Sweden  
TU Vienna, Vienna, Austria  
Ufa State Aviation Technical University, Ufa, Russia  
Universidad de Malaga UMAG, Malaga, Spain  
Universidade de Aveiro, Aveiro, Portugal  
Universität Leoben, Leoben, Austria  
Université Blaise Pascal-Clermont II, Aubière Cedex, France  
Université de Rouen, Rouen, France  
Universiteit Leiden, Leiden, The Netherlands  
University of Birmingham, Birmingham, UK  
University of California Santa Barbara, Santa Barbara, USA  
University of Cambridge, Cambridge, UK  
University of Edinburgh, Edinburgh, UK  
University of Greenwich, London, UK  
University of Leeds, Leeds, UK  
University of Liverpool, Liverpool, UK  
University of Lorena, Lorena, Brazil  
University of Lyon, Lyon, France



University of Science and Technology, Beijing, China  
University of St. Petersburg, St. Petersburg, Russia  
University of Vienna, Vienna, Austria  
University of Wales Swansea, Swansea, UK  
Uppsala University, Uppsala, Sweden  
Vanderbilt University, Nashville, USA  
Vrije Universiteit Brussel, Brussels, Belgium  
Xiamen-University, Xiamen, China  
Yarmouk University Irbid, Jordan

## Industrial Partners

### National:

Advalytix AG, Brunnthal  
ALD Vacuum Technologies AG, Hanau  
B. Laufenberg GmbH, Krefeld  
BASF AG, Ludwigshafen  
Biochem Zusatzstoffe Handels - und Produktionsgesellschaft mbH, Lohne  
BBT Thermotechnik GmbH, Wezlar  
Behr GmbH & Co. KG, Stuttgart  
Benteler Automobiltechnik GmbH, Paderborn  
Benteler Stahl & Rohr GmbH, Paderborn  
Bubbenzer Bremsen, Kirchen  
Buderus Edelstahl GmbH, Wetzlar  
CDP Bharat Forge GmbH, Ennepetal  
Chemetall GmbH, Frankfurt (Main)  
Cognis Deutschland GmbH & Co. KG, Monheim  
Continental AG, Hannover  
Daimler AG, Ulm  
Daimler AG, Stuttgart  
DILAS Diodenlaser GmbH, Mainz  
DYNAmore GmbH, Stuttgart  
Edelstahlwerke Buderus AG, Wetzlar  
EdgeWave GmbH, Würselen  
Georgsmarienhütte GmbH, Georgsmarienhütte  
GKS Gemeinschaftskraftwerk Schweinfurt GmbH, Schweinfurt  
H.C. Starck GmbH & Co. KG, Laufenburg  
Henkel KGaA, Düsseldorf



Hirschvogel Umformtechnik GmbH Gesenkschmiede und Preßwerk, Denklingen  
Honda R & D Europe, Offenbach (Main)  
Houghton GmbH, Aachen  
Hydro Aluminium Deutschland GmbH, Bonn  
Ingeneric GmbH, Aachen  
Inno-shape GmbH, Aachen  
IQ Evolution GmbH, Düsseldorf  
ISPAT GmbH, Duisburg  
Jenoptik Laserdiode GmbH, Jena  
Kirchhoff Automotive Deutschland GmbH, Attendorn  
Osram Opto Semiconductors GmbH, Regensburg  
Philips Technologie GmbH Business Center Automotive, Aachen  
Rasselstein GmbH, Andernach  
Robert Bosch GmbH, Stuttgart  
Salzgitter Mannesmann Forschung GmbH, Salzgitter  
Schweißtechnische Lehr- und Versuchsanstalt Halle GmbH, Halle (Saale)  
Siemens AG Corp. Tech., Düsseldorf  
SJM Co. Ltd., Ludwigshafen  
Termotek AG, Rastatt  
ThyssenKrupp Electrical Steel GmbH, Gelsenkirchen  
ThyssenKrupp Nirosta GmbH, Krefeld  
ThyssenKrupp VDM GmbH, Werdohl  
ThyssenKrupp Steel AG, Duisburg  
ThyssenKrupp Titanium, Essen  
TRW Automotive GmbH, Barsinghausen  
Vattenfall Europe AG, Berlin

**International:**

Acerinox SA Madrid, Madrid, Spain  
Afval Energie Bedrijf, Amsterdam, The Netherlands  
Arcelor Research SA, Puteaux, France  
Arcelor Research SA, Maizière-les-Metz, France  
ASM Brescia SPA, Brescia, Italy  
AVR - Afvalverwerking Rijnmond, Rozenburg, The Netherlands  
Bekaert, Zwevegem, Belgium  
Calcom ESI SA, Lausanne, Switzerland  
CNH Belgium N.V., Zedelgem, Belgium



CNR-IENI, Milano, Italy  
CNR-IENI-Genoa, Genova, Italy  
CORUS Technology B.V., IJmuiden, The Netherlands  
Corus Technology BV, Velsen Noord, The Netherlands  
Corus UK Ltd., London, UK  
Doncasters Group Ltd, Melbourne, Australia  
ELKEM Research, Kristiansand, Norway  
Galvalange SARL, Dudelange, Luxembourg  
Johnson Matthey Plc., London, UK  
Kema Nederland BV Arnhem, Arnhem, The Netherlands  
Keppel Seghers Belgium NW, Willebroek, Belgium  
NPL Management Ltd., Teddington, UK  
Oy Hydrocell Ltd., Järvenpää, Finland  
Rolls-Royce Plc., London, UK  
Soudage Automatique, Holtzwihr, France  
SSAB Tunnpilat AB, Borlänge, Sweden  
Surface specialities SA, Anderlecht, Belgium  
SVUM a.s. Prague, Prague, Czech Republic  
Thomas Swan Scientific Equipment Ltd, Cambridge, UK  
Top Analytica Ltd., TOPANALYT, Turku, Finland  
Trondheim Energiverk Fjernvarme AS, Trondheim, Norway  
voestalpine Stahl GmbH, Linz, Austria



# Patent

<b>Date of Issue</b>	<b>Description</b>	<b>Inventors</b>
2008-06-18	Beta-Titanlegierung, Verfahren zur Herstellung eines Warmwalzproduktes aus einer solchen Legierung und deren Verwendung EP 1 641 950 B1	Frommeyer, G., Prof. Dr. (20%) Knippscheer, S., Dr. (20%) Sibum, H., Dr. (40%), Deutsche Titan GmbH Schauerte, O., Dr. (20%), Volkswagen AG



# Conferences, Symposia and Meetings Organized by the Institute

## 2007

*M. Palm* organized and chaired the 15<sup>th</sup> meeting of the Fachausschuss Intermetallische Phasen (technical committee 'intermetallic phases') of the DGM at the MPIE on 2007-01-10.

*F. Stein* organized and chaired the International Workshop 'The Nature of Laves Phases VIII', which took place at the MPIE on 2007-01-11.

*L. Lympferakis* organized the NBN international workshop and PARSEM Midterm Meeting in Berlin from 2007-02-06 to 2007-02-09.

*J. Neugebauer and T. Hickel* were the organizers of the 'Focus meeting of the SPP1239: Fundamentals of the Magnetic Shape Memory Effect: Material properties & simulations', Ringberg, Kreuth from 2007-02-14 to 2007-02-16.

*A. W. Hassel* organized the 92. AGEF seminar 'Electrochemical Aspects of Nanowires and Nanotubes' at the MPIE on 2007-04-18.

*A. R. Pyszalla*, together with *A. Borbely* co-organized the MECASENS IV Conference in co-operation with the Institute for Material Science and Technology, TU Vienna, Vienna, Austria from 2007-09-24 to 2007-09-26.

*J. Neugebauer and O. Marquardt* co-organized the '2<sup>nd</sup> Harzer Ab Initio Workshop', Clausthal-Zellerfeld from 2007-10-14 to 2007-10-17.

*M. Palm, M. Spiegel, F. Stein and R. Krein* organized the '4<sup>th</sup> Discussion Meeting on the Development of Innovative Iron Aluminium Alloys', held at the Treff Metropole, Interlaken, Switzerland from 2007-10-23 to 2007-10-24.

*D. Raabe, F. Varnik and K. Berens* organized the International Max-Planck Workshop on 'Multiscale Modeling of Condensed Matter', Sant Feliu de Guixols, Spain from 2007-10-28 to 2007-11-02.

## 2008

*M. Palm* organized and chaired the 16<sup>th</sup> meeting of the Fachausschuss Intermetallische Phasen (technical committee 'intermetallic phases') of the DGM at MPI for Chemical Physics of Solids, Dresden on 2008-01-09.

*J. Neugebauer and M. Friák* organized the German-Korean miniworkshop, Düsseldorf on 2008-01-31.

*D. Raabe, F. Roters, P. Eisenlohr (MPIE), P. Gumbsch, H. Riedel and T. Hochrainer* organized the International Symposium on 'Computational Mechanics of Polycrystals', Bad Honnef from 2008-02-25 to 2008-02-26.

*J. Neugebauer*, together with *M. Asta* organized the Symposium 'Modern developments in multiphysics materials simulations' at the DPG Spring Meeting of the Condensed Matter Section, Berlin from 2008-02-25 to 2008-02-29.

*F. Roters* organized the Meeting of the Fachausschuss Computersimulation of the DGM entitled 'Anwendung der Simulation bei der Herstellung von Stahl -- Von der Schmelze zum Halbzeug' held at Böhler Edelstahl GmbH & Co KG in Kapfenberg, Austria from 2008-04-03 to 2008-04-04.

*A. R. Pyszalla* co-organized the 'International Symposium on Friction, Wear and Wear Protection', Aachen from 2008-04-09 to 2008-04-11.

*G. Frommeyer and F. Stein* together with the European Space Agency (ESA) and the German Aerospace Center (DLR) jointly organized a workshop entitled 'Space for Innovation: Industry Forum for Material Research and Microgravity' at the MPIE on 2008-05-08.

*A. Borbely* in co-operation with Eötvös University, Budapest, organized the Workshop 'On line-profile analysis', Budapest, Hungary from 2008-05-19 to 2008-05-20.



*J. Neugebauer* was the organizer of the Symposium G 'Wide band gap semiconductor nanostructures for optoelectronic applications', E-MRS Strasbourg, France from 2008-05-26 to 2008-05-30.

*A. W. Hassel* was co-organizer of the workshop 'Engineering of Functional Interfaces EnFI 2008', which took place in Jülich from 2008-06-12 to 2008-06-13.

*J. Neugebauer, M. Friák and T. Hickel* organized the International workshop on 'Ab Initio Description of Iron and Steel (ADIS2008): Magnetism and Phase diagrams', Schloss Ringberg, Kreuth from 2008-06-15 to 2008-06-20.

*F. Stein* organized and chaired the International Workshop 'The Nature of Laves Phases XI', which took place at the MPIE on 2008-07-17.

*J. Neugebauer, J. von Pezold and M. Todorova* were the organizers of the Computational Materials Science Workshop, Schloss Ebernburg, Bad Münster am Stein-Ebernburg from 2008-08-25 to 2008-08-29.

*A. R. Pyzalla* was a member of the Scientific Programme Committee for the Workshop on 'X-Ray Micro Imaging of Materials, Devices and Organisms', Dresden from 2008-10-22 to 2008-10-24.

*M. Palm* co-organized and chaired the Symposium 'Advanced Intermetallic-based Alloys for Extreme Environment and Energy Applications' at the MRS Fall Meeting, Boston, USA from 2008-12-01 to 2008-12-05.



# Institute Colloquia and Invited Seminar Lectures

## 2007

*P. Truman*, Leibniz-Institut für Polymerforschung Dresden e.V.: Separation of Incompatible Liquid Mixtures on Chemically Patterned Surfaces (2007-01-08).

*V. K. Pecharsky*, Iowa State University / Ames Laboratory, Ames, IA, USA: Research on Laves phases and Intermetallic Compounds at Ames Laboratory (2007-01-11, Colloquium).

*B. Jagoda-Cwiklik*, Institute of Organic Chemistry and Biochemistry of the Academy of Sciences of the Czech Republic in Prague: Theoretical study of 1) n-butane in silicalite-1 and 2) ion pairs in water (2007-01-15).

*L. Cwiklik*, Institute of Organic Chemistry and Biochemistry of the Academy of Sciences of the Czech Republic in Prague: Structure and reactivity of solid and liquid surfaces (2007-01-15).

*R. Bormann*, Technische Universität Hamburg-Harburg: Novel Nanostructured Hydride Composites: Hydrogen Storage Materials for Zero-Emission Car (2007-01-16, Colloquium).

*R. Besseling*, School of Physics, University of Edinburgh, UK: Imaging the Rheology of Colloidal Glasses: Shear Banding, Yielding and Microscopic Relaxation (2007-01-22).

*I. Doghri*, Université catholique de Louvain, Louvain la Neuve, Belgium: Micromechanics of Inelastic Composites: Modeling and Computation (2007-01-22).

*D. Rafaja*, Technische Universität Freiberg: Structure Research on Nanocomposites – Quo Vadis Structure Analysis (2007-02-06, Colloquium).

*F. Koenemann*, RWTH Aachen (peregrine systems): An Approach to Elastic Deformation Based on Thermodynamic Principles (2007-02-07).

*Y. Saito*, Waseda University, Tokyo, Japan: Modelling of Microstructural Evolutions in Structural Materials (2007-02-22).

*L. Delannay*, CESAME – MEMA Université catholique de Louvain, Louvain la Neuve, Belgium: Multisite Modelling of Grain Interactions in Single- and Multiphase Polycrystalline Aggregates (2007-03-05, Colloquium).

*P. Maugis*, Arcelor, Maizières-les-Metz, France: Precipitation Kinetics in Steel: From Atomistic Modelling to Analytic Laws (2007-03-21).

*I. Egry*, German Aerospace Center (DLR), Cologne: Structure and Properties of Liquid Metals (2007-04-07, Colloquium).

*P. Neff*, Technische Universität Darmstadt: Ein Cosserat-Modell zur Beschreibung von Kristallrotationen (2007-04-16).

*H. Vehoff*, Universität des Saarlandes: FIB-Tomography, Environmental NI-AFM and Orientation Gradient Mapping – New Techniques to Study Deformation, Fracture and Hydrogen Embrittlement Locally (2007-04-17).

*F. Körmann*, Humboldt-Universität zu Berlin: Temperature Driven Spin Reorientation Transition in Thin Films (2007-04-18).

*A. Udyansky*, MPI für Metallforschung, Stuttgart: Concentration fluctuations in binary alloys (2007-04-24).

*E. T. Lilleodden*, GKSS Forschungszentrum, Geesthacht: Size Effects in Metal Plasticity (2007-04-25).

*J. W. House*, Eglin AFB, USA: Thermo-Mechanical Processing of Tantalum and Zirconium for High Strain Rate Applications (2007-05-03).

*T. Ladd*, University of Florida, Gainesville, USA: Polymer Migration in Confined Geometries (2007-06-04, Colloquium).

*K. Lu*, Shenyang National Laboratory for Materials Science, Chinese Academy of Sciences, Shenyang, China: Strengthening Copper by Means of Nano-Scale Twins (2007-06-11).



*M. Militzer*, Centre for Metallurgical Process Engineering, Vancouver, Canada: Design of Modern Steels with Complex Fine-Grained Microstructure (2007-07-11, Colloquium).

*B. Aichmayer*, MPI for Colloids and Interfaces, Golm: The Effects of Organic Molecules on the Growth and Structure of Inorganic Crystals (2007-07-11).

*K.-U. Neumann*, Loughborough University, Leicestershire, UK: Neutron Scattering Investigations of the Ferromagnetic Shape Memory Compound  $\text{Ni}_2\text{MnGa}$  and Related Alloys (2007-07-17, Colloquium).

*J. Ireta*, Fritz-Haber-Institut Berlin: Description of the Protein Conformational Space using Density Functional Theory (2007-08-07).

*K. S. Kumar*, Brown University, Providence, USA: High Strain Rate Deformation and Microstructure Evolution in Ultra-High Strength Steels (2007-08-14, Colloquium).

*R. Drautz*, University of Oxford, UK: Development of Bond-Order Potentials for Bridging the Electronic to Microstructural Modelling Hierarchies in Materials Science (2007-08-14).

*A. Al-Sharif*, Yarmouk-University, Jordan: On the KS momentum density and atomic Compton profiles (2007-08-27).

*T. Eberlein*, University of Exeter, UK: Pointdefects in GaN and Rod-like Defects in Silicon (2007-09-07).

*M. Sluiter*, Delft University of Technology, The Netherlands: Alloy Phase Stability from First Principles (2007-10-09, Colloquium).

*R. Janisch*, Friedrich-Alexander-Universität Erlangen-Nürnberg: Ab initio investigations of structural and mechanical properties of interfaces (2007-11-12).

*M. Havenith-Neven*, Ruhr-Universität Bochum: A Closer Look on Surfaces and Interfaces by IR- and THz Laser Spectroscopy (2007-11-13, Colloquium).

*A. Hartmaier*, Friedrich-Alexander-Universität Erlangen-Nürnberg: Large-Scale Atomistic Simulations of Nanoindentation: Dislocation Microstructure and Indentation Size Effect (2007-11-13).

*S. Ishikawa*, Tokyo Institute of Technology, Japan: Phase Equilibria between  $\gamma$ -Fe and  $\text{Fe}_2(\text{Mo},\text{Nb})$  Laves phases and the Phase Stability of Laves phases with C14 and C15 Structures (2007-11-14).

*C. G. van de Walle*, University of California, Santa Barbara, USA: Role of Defects in Kinetics of Hydrogen Storage Materials (2007-11-19).

*U. Naumann*, RWTH Aachen: LuFG Software and Tools for Computational Engineering: Adjoint Code Compilers (2007-12-05).

## 2008

*K. Wandelt*, Universität Bonn: Trends in Surface Science (2008-01-15, Colloquium).

*W. Petry*, Technische Universität München, Munich: Neutrons for Material Sciences and Phonons at Martensitic Phase Transitions (2008-01-17, Colloquium).

*M. Acet*, Universität Duisburg-Essen: Magnetic Shape-Memory and Magnetocaloric Effects Close to First Order Transitions (2008-01-22, Colloquium).

*S. Hild*, Universität Ulm: Terrestrial Isopods: A Model System to Study Formation and Function of Amorphous Calcium Carbonate in Biocomposites (2008-01-24).

*G. Pfanner*, Universität Graz, Austria: A study on photon-entanglement from the biexciton cascade in a quantum dot (2008-01-28).

*V. Heine*, Cavendish Laboratory, University of Cambridge, UK: The theoretical chemistry of inter-atomic bonding in metals (2008-01-29).

*B. Lange*, Universität Paderborn: Ammonia adsorption on  $\text{Si}(001):\text{Cl}$  - First-principles calculations (2008-02-08).

*J. G. Swadener*, Los Alamos National Laboratory, Los Alamos, USA: Some Interrelationships between Stress and Biological Materials (2008-02-18).



- J. G. Swadener*, Los Alamos National Laboratory, Los Alamos, USA: Strain, Dislocations and Functional Property Modifications in Si/Ge Nanowires (2008-02-19).
- G. Scalia*, MPI für Festkörperforschung, Stuttgart: Liquid Crystals and Carbon Nanotubes: Anisotropic Materials for Functional Composites (2008-02-22).
- G. Moras*, King's College London, UK: Molecular dynamics simulations of 'Smart Cut<sup>®</sup>' cleavage of silicon devices through a novel 'Learn On The Fly' hybrid quantum/classical formalism (2008-03-04).
- F. Barlat*, Alcoa Technical Center, Pennsylvania, USA: Modeling Planestrain Fracture Toughness in 7085 Aluminium Alloy (2008-03-07).
- R. Kirchheim*, Universität Göttingen: A Novel Way of Describing the Interaction of Solute Atoms and Defects (2008-03-18, Colloquium).
- P. Dondl*, MPI für Mathematik in den Naturwissenschaften, Leipzig: Depinning Behavior of Martensitic Interfaces in Heterogeneous Media (2008-03-20).
- V. Vovk*, University of Oxford, UK: Thermal Stability of Co/Cu and Py/Cu GMR Sensor Materials (2008-04-07).
- G. Wittstock*, Universität Oldenburg: Scanning Electrochemical Microscopy for Kinetic Characterisation and Local Modification of New Materials and Functional Layers (2008-04-08, Colloquium).
- T. Sontheimer*, RWTH Aachen and Harvard University, Cambridge, USA: Crystallization Kinetics in Amorphous Tellurium Alloys used for Phase Change Recording (2008-04-15).
- V. L. Welch*, University of Namur (FUNDP), Belgium: From Comb-Jellyfish to Rainforest Weevils: The Problem of Producing Colouration and Nature's Elegant Solutions (2008-04-21).
- W. Skrotzki*, Technische Universität Dresden: High Strain Torsion of NiAl (2008-05-06, Colloquium).
- P. Schaffnit*, ACCESS, RWTH Aachen: Phase Field Modelling (2008-05-07).
- J. Szeszko*, Unipress, Polish Academy of Sciences, Warsaw, Poland: Luminescence in InGaN/GaN Quantum Structures (2008-05-15).
- P. Rinke*, University of California, Santa Barbara, USA: Defect Formation Energies without the Band Gap Problem - Combining DFT and GW for the Silicon Self-Interstitial (2008-05-27).
- J. Hald*, University of Denmark, Lyngby, Denmark: Martensitic 9-12%Cr Steels for Application in Advanced Power Plants - Development Status and Future Prospects (2008-06-03, Colloquium).
- D. Quéré*, ESPCI, Paris, France: Wetting and Roughness (2008-06-10, Colloquium).
- T. Maki*, Kyoto University, Japan: Development of Thermomechanical Processing of Steels (2008-06-23, Colloquium).
- H. Sturm*, Federal Institute for Materials Research and Testing (BAM), Berlin: Modulated Lateral Force Microscopy: An AFM Tool for Analysis and Modification of Polymer Surfaces (2008-07-03).
- M. and B. Lenz* (winner of the German 'Jugend forscht' award 2007), Enkirch: Untersuchungen zum 'Saitensprung' (2008-07-09).
- A. Erbe*, Nanobioscience Group, Academia Sinica, Taipei, Taiwan: Funny games with light at interfaces, The interface of colloids, redoxactive proteins in lipid bilayers and submicrometer channels (2007-07-09).
- T. Ungár*, Eötvös University Budapest, Hungary: Twinning, Slip Activity and Dislocation Densities Determined in Hexagonal Materials by the Method of X-Ray Line Profile Analysis (2008-07-10, Colloquium).
- P.-P. Choi*, Korea Institute of Science and Technology, Seoul, South-Korea: Nanoscale Materials Characterization using Atom Probe Tomography (2008-07-15, Colloquium).
- A. Gurlo*, Darmstadt University of Technology: Fundamental studies of gas sensing mechanism on semiconducting metal oxides (2008-07-17).
- M. A. Crimp*, Michigan State University, East Lansing, USA: Microscale Characterization in the SEM: Electron Channeling and Diffraction for Orientation Analysis and Defect Imaging (2008-07-18).
- J. Fick*, The University of Maine, Orono, USA: Vibrational Sum-Frequency Generation at Self-Assembled Monolayers of Ethylene Glycols and Lipid Bilayers (2008-07-18).



- C. Loschen*, Philips- Universität Marburg: Theoretical Studies of Bulk and Nanoscale Cerium Oxides (2008-07-24).
- M. J. R. Sandim*, Universidade de São Paulo, Lorena, Brazil: Annealing Effects on the Microstructure and Magnetic Properties of Cu-Nb Composites (2008-08-06).
- A. Bund*, TU Dresden: Characterization and modeling of electrochemical interfaces at small length scales (2008-08-08).
- V. Vitek*, University of Pennsylvania, Philadelphia, USA: Multiscale Modeling of Plastic Deformation of Transition BCC Metals from Atomic to Continuum Level (2008-08-12, Colloquium).
- N. Tsuji*, Osaka University, Japan: Managing Both High Strength and Ductility in Ultrafine Grained Steels (2008-08-15).
- B. Eidel*, Technische Universität Darmstadt: A Framework for Atomistic-Continuum-Coupling Based on Energy-Sampling in Clusters and Its Application to Nanoindentation (2008-08-25).
- H. Ding*, Northeastern University, Shenyang, China: Experimental Investigation and FEM-Simulation on ECAP of High Mn Steel (2008-08-28).
- Y. Adachi*, National Institute for Materials Science (NIMS), Tsukuba, Japan: 2D/3D/4D EBSD-Neutron Study on Ferrite, Pearlite, Bainite, Martensite and Austenite in Steels (2008-09-09).
- T. Niedermayer*, Universität Marburg: Modeling Synchronization, Phase Locking and Metachronal Waves in Ciliary Chains (2008-09-09).
- E. Monaco*, TU Clausthal: Lattice Boltzmann Simulations of Multiphase Flows at High Density Ratios (2008-09-10).
- R. Nazarov*, Mining School of Nancy and Moscow Institute of Steels and Alloys, Russia: Identification of mechanical behavior law in relation with material properties for metal forming (2008-09-16).
- C. W. Sinclair*, University of British Columbia, Vancouver, Canada: Strain Induced Martensitic Transformations: Experiments and Simulation (2008-09-18).
- C. Chou*, Institute of Physics, Academia Sinica, Taiwan: The Magical World of Micro-to-Nanofluidic Interface and the Extremely Confined Environment for Molecular/Cellular Analysis (2008-09-23).
- F. Dörfler*, MPI für Metallforschung, Stuttgart: Wetting on chemical channels: liquid morphologies in the micro and nano world (2008-09-24).
- J. Neuhaus*, TU München, Munich, Forschungsreaktor Munich II: Phonons and martensitic transformations (2008-10-10).
- U. Kamachi Mudali*, Homi Bhabha National Institute, Indira Gandhi Centre for Atomic Research, Kalpakkam, Mumbai, India: Indira Gandhi Centre for Atomic Research and Fast Breeder Reactor Programme OR (2008-10-10).
- D. Zahn*, MPI für Chemische Physik fester Stoffe, Dresden: Atomistic Simulation Approaches to Structural Transformations and Self-Organization (2008-10-13, Colloquium).
- D. Cédât*, École Centrale Paris, France: Experimental characterization and mechanical behaviour modelling of Mo-TiC composite for high temperature applications (2008-10-16).
- J. Schwartz*, École Centrale Paris, France: Fatigue initiation modelling of 316L and IF steel based on the non local plasticity theory (2008-10-16).
- R. Spolenak*, Materials Research Center (MRC) ETH Zurich, Switzerland: Mechanical Properties of Metals at the Nanometer Scale: From Particles to Wires, Thin Films and 3D Structures (2008-10-20, Colloquium).
- H. van Swygenhoven-Moens*, Paul-Scherrer Institute, Materials Science & Simulation, Villigen, Switzerland: Small Scale Plasticity (2008-10-21, Colloquium).
- E. Eggeling*, TU Graz, Austria: Modelling and simulation of grain growth (2008-10-22).
- R. Busch*, Universität des Saarlandes: Bulk Metallic Glass: A New Engineering Material (2008-11-03, Colloquium).



A. *Ludwig*, Ruhr-Universität Bochum: Development of Multifunctional Thin Films using High-Throughput Experimentation and MEMS (2008-11-05, Colloquium).

D. *Weygand*, University of Karlsruhe, izbs, Karlsruhe: Plasticity of Micro-Samples: A Discrete Dislocation Dynamics Study (2008-11-07).

Y. *Kobayashi*, Tokyo Institute of Technology, Tokyo, Japan: Inverse-utilization of impurities in steel production (2008-11-17).

J. *Konrad*, Salzgitter Mannesmann Forschung GmbH, Duisburg: Innovative Development of new Steel Grades for Pipes and Tubes (2008-11-25).

A. *Meyer*, German Aerospace Center (DLR), Cologne: Atomic Transport in Liquid Alloys (2008-12-09, Colloquium).



# Lectures and Teaching at University

## 2007

*G. Frommeyer*, TU Clausthal: Leichtbauwerkstoffe auf der Basis intermetallischer Phasenlegierungen, WS 2006/2007, WS 2007/2008.

*G. Frommeyer*, TU Clausthal: Hochfeste und supraduktile Leichtbaustähle, SS 2007.

*A. W. Hassel*, Ruhr-Universität Bochum: Introduction into physical chemistry of surfaces and interfaces, WS 2007/2008.

*A. W. Hassel*, Ruhr-Universität Bochum: Laboratory course on surface spectroscopy and microscopy, WS 2007/2008.

*A. W. Hassel*, Ruhr-Universität Bochum: Reactions at surfaces and interfaces I: Electron Transfer Reactions, WS 2007/2008.

*J. Neugebauer*, Universität Paderborn: Computational Material Science (Practicum) WS 2006/2007.

*D. Raabe*: Crystallographic Textures, MPIE, Düsseldorf, Lecture Series – Lectures in SFB 761 ‚Stahl ab initio‘ Measurement, microstructure analysis, coupling micromechanical mechanisms, 2007-06-09.

*M. Rohwerder*, Ruhr-Universität Bochum, Fakultät für Maschinenbau, Institut für Werkstoffe: Funktionelle Schichten auf Metallen: vom Korrosionsschutz zum Lotus-Effekt, SS 2007.

*M. Rohwerder*, Ruhr-Universität Bochum, Fakultät für Maschinenbau, Institut für Werkstoffe: Surface and interface spectroscopy and microscopy, WS 2007/2008.

*M. Rohwerder*, Ruhr-Universität Bochum, Fakultät für Maschinenbau, Institut für Werkstoffe: Lab course on surface spectroscopy and microscopy, WS 2007/2008.

*M. Rohwerder and M. Stratmann*, Ruhr-Universität Bochum, Fakultät für Maschinenbau, Institut für Werkstoffe: Korrosion, SS 2007.

*F. Roters*, RWTH Aachen: Prozess- und Werkstoffsimulation, WS 2006/2007.

*F. Varnik and D. Raabe*, RWTH Aachen: Lattice Boltzmann simulation of non-ideal fluids, WS 2007/2008.

*M. Winning*, RWTH Aachen: Theoretische Metallkunde II, SS 2007.

*S. Zaeferrer*, MPIE Düsseldorf: Lecture for International Max-Planck Research School, SurMat Analytical transmission and scanning electron microscopy, 2007-03-22 to 2007-03-23.

## 2008

*W. A. Counts*, RWTH Aachen, EU Regional School – Materials Science in Aachen Institute for Advanced Studies in Computational Engineering and Science (AICES): FEM: A Basic Overview of the Method & Outlook on Applications. EU-Regional School Trimester III/2008.

*P. Eisenlohr*, RWTH Aachen: EU Regional School – Materials Science in Aachen Institute for Advanced study in Computational Engineering Science (AICES): Theory and Applications of a Computational Framework for Crystal Plasticity. 2008-08-04 to 2008-09-22.

*A. Erbe, together with M. Havenith and C. Wöll*, Ruhr-Universität Bochum: Spektroskopie an Oberflächen, WS 2008/2009.

*M. Friák*, RWTH Aachen, EU Regional School – Materials Science in Aachen Institute for Advanced study in Computational Engineering Science (AICES): Ab initio based approaches to the theory-guided materials design, 2008-09-22.

*G. Frommeyer*, TU Clausthal: Hochfeste und supraduktile Leichtbaustähle, SS 2008.

*G. Frommeyer*, TU Clausthal: Leichtbauwerkstoffe auf der Basis intermetallischer Phasenlegierungen, WS 2008/2009.



- G. Frommeyer*, Stahl Akademie, Krefeld: Aufbaustudium für Führungskräfte: Entwicklung und Eigenschaften der TWIP Stähle, 2008-09-11.
- A. W. Hassel*, Ruhr-Universität Bochum: Introduction into physical chemistry of surfaces and interfaces, WS 2007/2008.
- A. W. Hassel*, Ruhr-Universität Bochum: Laboratory course on surface spectroscopy and microscopy, WS 2007/2008.
- A. W. Hassel*, Ruhr-Universität Bochum, Reactions at surfaces and interfaces I: Electron Transfer Reactions, WS 2007/2008.
- A. W. Hassel and W. Schuhmann*, Ruhr-Universität Bochum: Bioanalytik und Sensorik, SS 2008.
- D. Raabe*, RWTH Aachen: ‚Die Geschichte der Werkstoffe‘ (History of Metallurgy) in der RWTH Lecture, Einführung in die Werkstofftechnik, SS 2008.
- D. Raabe*, RWTH Aachen Micromechanics of Materials (Lecture 3 and Exersize 1), (Master classes in English language), SS 2008.
- M. Rohwerder*, Ruhr-Universität Bochum, Fakultät für Maschinenbau, Institut für Werkstoffe: Funktionelle Schichten auf Metallen: vom Korrosionsschutz zum Lotus-Effekt, SS 2008.
- F. Roters*, RWTH Aachen: Prozess- und Werkstoffsimulation, WS 2007/2008.
- M. Winning*, RWTH Aachen: Theoretische Metallkunde II, SS 2008.
- F. Varnik and D. Raabe*, RWTH Aachen: Computer aided modelling of wetting phenomena: Theory and application, SS 2008.
- F. Varnik and D. Raabe*, RWTH Aachen: Lattice Boltzmann simulation of non-ideal fluids, WS 2007/2008.
- S. Zaeferrer*, Canadian Microscopical Society, Montreal, Canada: Workshop on the electron backscatter diffraction (EBSD) technique – fundamentals and applications, 2008-05-20.



# Oral and Poster Presentations

## 2007

*Abu-Farsakh, H., J. Neugebauer and M. Albrecht:* Ab-initio Studies on Alloy Formation in Diluted Nitrides. (Workshop on Nitride Based Nanostructures, 2007-02-05 to 2007-02-07, Berlin).

*Abu-Farsakh, H. and J. Neugebauer:* In-N anti-correlation in InGaAsN alloys: the delicate interplay between adatom thermodynamics and kinetics. (Spring meeting of the German Physical Society (DPG), 2007-03-26 to 2007-03-30, Regensburg).

*Abu-Farsakh, H., J. Neugebauer and M. Albrecht:* Ab-initio study of compositional anti-correlation of In and N in InGaAsN alloys. (The 7<sup>th</sup> International Conference of Nitride Semiconductors (ICNS-7), 2007-09-16 to 2007-09-21, Las Vegas, USA).

*Abu-Farsakh, H. and J. Neugebauer:* Ab-initio study of the thermodynamics and kinetics of N at GaAs(001) surface. (PAW workshop 2007, 2007-10-15 to 2007-10-17, Goslar).

*Agudo, L., A. Kostka, S. Weber, J. Wagner, E. Arenholz, J. Bruckner and A. R. Pyszalla:* Aluminium-Rich  $Fe_xAl_y(Si_z)$  Phase Formation in Al/Steel Welds with AISi Fillers. (Euromat 2007, 2007-09-10 to 2007-09-13, Nürnberg).

*Agudo, L., S. Weber, J. Wagner, E. Arenholz, J. Bruckner and A. R. Pyszalla:* Intermetallic  $Fe_xAl_y$ -Phases in Steel/Al-Alloy Fusion Welds. (Euromat 2007, 2007-09-10 to 2007-09-13, Nürnberg).

*Agudo, L., A. Kostka, S. Weber, J. Wagner, E. Arenholz, J. Bruckner and A. R. Pyszalla:* Al/Steel welds: Fundamental investigation of intermetallic phases. (4<sup>th</sup> Discussion Meeting on the Development of Innovative Iron Aluminium Alloys, FeAl2007, 2007-09-22 to 2007-09-24, Interlaken, Switzerland).

*Agudo, L., H. Pinto, A. Kostka, S. Weber, J. Wagner, E. Arenholz, J. Bruckner and A. R. Pyszalla:* Study of Microstructure and Residual Stresses in Dissimilar Al/Steel Welds Produced by Cold Metal Transfer. (MECASSENS IV, 2007-09-24 to 2007-09-26, Vienna, Austria).

*Balasundaram, K., G. Grundmeier and D. Raabe:* Nanomechanics of thin glassy polymer films. (3<sup>rd</sup> International Indentation Workshop, 2007-07-15 to 2007-07-20, Cavendish Laboratory, University of Cambridge, Cambridge, UK).

*Balasundaram, K., G. Grundmeier and D. Raabe:* Nanomechanics of thin glassy polystyrene films. (1<sup>st</sup> IMPRS-SurMat Workshop on Surface and Interface Engineering in Advanced Materials, 2007-08-15, Klosterhost).

*Barbatti, C., J. Garcia, G. Liedl, H. Pinto and A. R. Pyszalla:* Joining of Hardmetal to Steel by Laser Beam Welding. (Verbundwerkstoffe und Werkstoffverbunde, 2007-03-14 to 2007-03-16, Bremen).

*Barbatti, C., J. Garcia and A. R. Pyszalla:* Development and Characterization of PM Components with Optimum Properties. (PM Training Course, 2007-06-23 to 2007-07-01, Kosice, Slovakia).

*Barbatti, C., F. Sket, A. di Prinzio, M. Staia, J. Garcia and A. R. Pyszalla:* Influence of Binder Composition, Nitridation and Treatment of Coatings on the Microstructure, Corrosion and Wear of (W,Ti)C-(Ta,Nb)C-Co/Fi/Fe Hardmetals. (PM Training Course, 2007-06-23 to 2007-07-01, Kosice, Slovakia).

*Barbatti, C., H. Pinto, A. di Prinzio, M. Staia, R. Pitonak, J. Garcia and A. R. Pyszalla:* Influence of Microblasting on the Microstructure and Residual Stresses of CVD k- $Al_2O_3$  Coated Hardmetals. (EUROMAT 2007, 2007-09-10 to 2007-09-13, Nürnberg).

*Barbatti, C., H. Pinto, A. di Prinzio, M. Staia, R. Pitonak, J. Garcia and A. R. Pyszalla:* Influence of Microblasting on the Microstructure and Residual Stresses of CVD k- $Al_2O_3$  Coated Hardmetals. (MECASSENS IV, 2007-09-24 to 2007-09-26, Vienna, Austria).

*Bastos, A., N. Zaafarani, S. Zaefferer and D. Raabe:* Overview on 3D EBSD. (Deutsche Gesellschaft für Materialkunde e.V., Fachausschuss Texturen, 2007-03-23, RWTH Aachen, Aachen).

*Bastos, A., S. Zaefferer and D. Raabe:* 3D Orientation microscopy. (Fachausschuss Texturen, 2007-03-23, RWTH Aachen, Aachen).

*Bastos, A., S. Zaefferer and D. Raabe:* 3 Dimensional EBSD study of the relationship between triple junctions and columnar grain in electrodeposited materials. (Electron Back Scatter Diffraction Meeting 2007, 2007-03-26 to 2007-03-28, New Lanark, UK).

*Bastos, A., S. Zaefferer and D. Raabe (invited):* 3D Orientation microscopy. (1<sup>st</sup> Annual EDAX EBSD German User's Group Meeting, 2007-06-05, Wiesbaden).

*Bastos, A., S. Zaefferer, D. Raabe:* Microstructure evolution during electrodeposits film growth. (EUROMAT 2007, 2007-09-10 to 2007-10-13, Nürnberg).



*Bergman, T., P.-E. Sundell, P. Keil, G. Grundmeier, M. Schatzl, M. Heylen, K. Sundberg, A. Alanen, M. Rosenberg, K. Laurila, J. Lamminmäki and L.-M. Äikäs:* UV Coil coating - importance of thermal treatment. (Radtech Europe, 2007-11-13 to 2007-11-15, Vienna, Austria).

*Biedermann, P., E. Torres and A. Blumenau:* Oxygen Reduction at Thiol/Au(111)SAMs, Atomistic Modelling and Experiment. (212<sup>th</sup> ECS Meeting, 2007-10-07 to 2007-10-12, Washington (DC), USA).

*Biedermann, P. U., E. Torres, L. Laaboudi, S. Isik-Uppenkamp, M. Rohwerder and A. T. Blumenau:* Cathodic Delamination by a Combined Computational and Experimental Approach: The Alkylthiol/Gold Model System. (Multiscale Material Modeling of Condensed Matter, MMM2007, 2007-10-28 to 2007-11-02, Sant Feliu de Guixols, Spain).

*Biedermann, P. U. and A. T. Blumenau:* Ab-Initio Calculation of the Standard Hydrogen Electrode Potential and Application to the Mechanism of the Oxygen Reduction. (Workshop on Converging Theoretical and Experimental Approaches to Corrosion, 2007-11-07, MPIE, Düsseldorf).

*Blumenau, A. T.:* Extended defects in GaN from an atomistic modelling point view. (OPTO 2007, Integrated Optoelectronic Devices, 2007-01-20 to 2007-01-25, San Jose, USA).

*Blumenau, A. T., P. U. Biedermann and E. Torres:* Modelling adhesion and delamination at oxide/polymer interfaces. (Multiscale Material Modeling of Condensed Matter, MMM2007, 2007-10-28 to 2007-11-02, Sant Feliu de Guixols, Spain).

*Blum, W., P. Eisenlohr, D. Amberger, K. Mili and M. Göken:* Microstructure–plasticity relationship of {Mg} -alloys at elevated temperatures. (100<sup>th</sup> Eastern Forum of Science and Technology 'Adv. Magnesium Alloys and Their Applications', 2007-08-01 to 2007-08-04, Shanghai, China).

*Borissov, D. and M. Rohwerder:* Fundamental Investigation of the Effect of the Oxides on the Reaction Kinetics during Hot Dip Galvanizing. (GALVATECH '07, 7<sup>th</sup> International Conference on Zinc and Zinc Alloy Coated Steel Sheet, 2007-11-18 to 2007-11-22, Osaka, Japan).

*Borodin and M. Rohwerder:* STM-investigation of self-assembly of phosphonates on model oxides. (ECASIA 2007, 12<sup>th</sup> European Conference on Applications of Surface and Interface Analysis, 2007-09-09 to 2007-09-14, Brussels-Flagey, Belgium).

*Brito, P., H. Pinto, M. Spiegel and A. R. Pyzalla:* Residual Stresses and Phase Composition in Oxide Layers grown on Iron Aluminides. (MECASSENS IV, 2007-09-24 to 2007-09-26, Vienna, Austria).

*Brito, P., H. Pinto, A. R. Pyzalla and M. Spiegel:* Phase composition and internal stress development during the oxidation of iron aluminides. (Final Conference COST Action 535 Thermodynamics of Alloyed Aluminides (THALU) and 4<sup>th</sup> Discussion Meeting on the Development of Innovative Iron Aluminium Alloys, 2007-10-21 to 2007-10-24, Interlaken, Switzerland).

*Brokmeier, K., G. Frommeyer and R. Rablbauer:* Mechanical properties of high carbon lightweight Mn-Al-Si TRIP/TWIP steels. (EUROMAT 2007, 2007-09-10 to 2007-09-13, Nürnberg).

*Bruder, K., A. W. Hassel, B. Mildner and D. Dising:* Impedance spectroscopy of thin (d = 4 nm) tantalum oxide films: Temperature and Field dependence. (Spring Meeting of the German Physical Society (DPG), 2007-03-26 to 2007-03-30, Regensburg).

*Bruder, K., A. W. Hassel, B. Mildner and D. Dising:* Impedance spectroscopy of thin (d = 4 nm) tantalum oxide films. (7<sup>th</sup> International Symposium on Electrochemical Impedance Spectroscopy, 2007-06-03 to 2007-06-08, Argelès-sur-Mer, France).

*Bruder, K., D. Dising and A. W. Hassel:* Electronic tunneling through anodic oxide barriers. (58<sup>th</sup> Annual Meeting of the International Society of Electrochemistry, 2007-09-09 to 2007-09-14, Banff, Canada).

*Bruder, K. and A. W. Hassel:* In-situ monitoring of the cleaning process of steel sheet. (GDCh Wissenschaftsforum 2007, 2007-09-16 to 2007-09-19, Ulm).

*Brunetti, G., R. Krein, T. Gossmidier and M. Palm:* Evaluation of the Brittle-to-Ductile Transition Temperature (BDTT) and the fracture modes in Fe-Al-X alloys. (4<sup>th</sup> Discussion Meeting on the Development of Innovative Iron Aluminium Alloys, 2007-10-21 to 2007-10-24, Interlaken, Switzerland).

*Calcagnotto, M., D. Ponge and D. Raabe:* Ultrafine grained dual phase steels. (GLADD Meeting, 2007-10-05, Aachen).

*Calcagnotto, M., D. Ponge and D. Raabe:* Fabrication of ultrafine grained dual phase steels. (National Institute for Materials Science (NIMS), 2007-10-22, Tsukuba, Japan).

*Calcagnotto, M., D. Ponge and D. Raabe:* Fabrication of Ultrafine Grained Ferrite/Martensite Dual Phase Steel by Large Strain Warm Deformation and Subsequent Intercritical Annealing. (ISUGS 2007 (International Symposium on Ultrafine Grained Steels), 2007-10-24 to 2007-10-26, Kitakyushu, Japan).

*Chen, Y., S. Milenkovic and A. W. Hassel:* Gold Nanostructures through Directional Eutectoid Transformation. (92. AGEF-Seminar DFG-SPP 1165: Elektrochemische Aspekte von Nanodrähten und Nanoröhren, 2007-04-18, Düsseldorf).



*Chen, Y., S. Milenkovic and A. W. Hassel:* Gold Nanostructures through directional Eutectoid Transformation. (EUROMAT 2007, European Congress and Exhibition on Advanced Materials and Processes, 2007-09-10 to 2007-09-13, Nürnberg).

*Coelho, R. S., A. Kostka, S. Riekehr, R. Zettler, S. Sheikhi, J. Dos Santos and A. R. Pyszalla:* Joining of light-weight materials by laser beam welding and friction stir welding. (2<sup>nd</sup> Alban Conference, 2007-05-12, Grenoble, France).

*Coelho, R., A. Kostka, H. Pinto, M. Kocak and A. R. Pyszalla:* Mikrostruktur, Textur und Eigenspannungen in Schweißverbindungen aus AZ31. (FA 13 Eigenspannungen der AWT, Forschungsgemeinschaft Werkzeuge und Werkstoffe e.V., 2007-06-06, Remscheid).

*Coelho, R. S., A. Kostka, S. Riekehr, M. Kocak and A. R. Pyszalla:* Microstructure and Properties of AZ31B Magnesium Alloy Laser Beam Welds. (Euromat 2007, 2007-09-10 to 2007-09-13, Nürnberg).

*Coelho, R. S., A. Kostka, R. Zettler, J. dos Santos and A. R. Pyszalla:* On the microstructure and residual stresses in friction-stir dissimilar AA6040 aluminium alloy to AZ31 magnesium alloy weld. (MECASSENS IV, 2007-09-24 to 2007-09-26, Vienna, Austria).

*Coelho, R., S., A. Kostka, S. Riekehr, M. Kocak and A. R. Pyszalla:* Microstructure and residual stress of Nd:YAG laser AZ31B butt welds. (MECASSENS IV, 2007-09-24 to 2007-09-26, Vienna, Austria).

*Coelho, R. S., A. Kostka, S. Sheikhi, J. dos Santos and A. R. Pyszalla:* Friction Stir Welding of ZStE340 High-strength Steel and AA6181-T4 Aluminium Alloy. (SOLVSTIR - International Seminar on Friction Stir Welding of Steels, 2007-11-22 to 2007-11-23, Geesthacht).

*Degees, J. R. Rablbauer and G. Frommeyer:* Mechanical properties of macroalloyed single phase Fe<sub>3</sub>Al base alloys of DO<sub>3</sub> order. (EUROMAT 2007, 2007-09-10 to 2007-09-13, Nürnberg).

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*Mardare, A. I., A. D. Wieck and A. W. Hassel:* High throughput processing and characterization of surface oxides using an automated scanning droplet cell. (212<sup>th</sup> ECS Meeting, 2007-10-07 to 2007-10-12, Washington (DC), USA).

*Marquardt, O., T. Hickel and J. Neugebauer:* A k.p-approach to electronic states and Coulomb interaction in semiconductor quantum dots. (Forschergruppentreffen Uni Bremen, 2007-01-16, Bremen).

*Marquardt, O., M. Wahn, L. Lympirakis, T. Hickel and J. Neugebauer:* Implementation and application of a multi-scale approach to electronic properties of group III-nitride based semiconductor nanostructures. (Workshop on Nitride Based Nanostructures, 2007-02-05 to 2007-02-09, Berlin).

*Marquardt, O., T. Hickel, B. Grabowski, S. Boeck and J. Neugebauer:* Implementation and application of the k.p-formalism to electronic structure and Coulomb matrix elements. (Spring meeting of the German Physical Society (DPG), 2007-03-26 to 2007-03-30, Regensburg).

*Marquardt, O., T. Hickel and J. Neugebauer:* Application of the 8-band k.p-formalism to semiconductor nanostructures. (Forschergruppentreffen Uni Bremen, 2007-05-21, Bremen).

*Marquardt, O., T. Hickel and J. Neugebauer:* A plane-wave approach to real-space properties of semiconductor nanostructures: Electronic states, strain profiles and piezoelectric fields. (PAW workshop 2007, 2007-10-15 to 2007-10-17, Goslar).

*Michalik, A., S. Isik-Uppenkamp, A. Laaboudi and M. Rohwerder:* Role of Localized Protection for the Performance of Conducting Polymer Based Composite Coatings. (212<sup>th</sup> ECS Meeting, 2007-10-07 to 2007-10-12, Washington (DC), USA).

*Milenkovic, S., A. J. Smith and A. W. Hassel:* Self-organised Metallic Nanostructures via Directional Solidification of Eutectics. (DFG-SPP 1165 Statusseminar, 2007, Fulda).

*Milenkovic, S. and A. W. Hassel:* Self-organised Metallic Nanostructures via Directional Solidification of Eutectics. (92. AGEF-Seminar DFG-SPP 1165: Elektrochemische Aspekte von Nanodrähten und Nanoröhren, 2007-04-18, Düsseldorf).

*Milenkovic, S. and A. W. Hassel:* Fabrication of self-organised metallic nanostructures. (Euronanoforum, 2007-06-19 to 2007-06-21, Düsseldorf).

*Milenkovic, S. and A. W. Hassel:* Gold nanostructures by directional solid-state decomposition. (2<sup>nd</sup> International Conference on Surfaces, Coatings and Nanostructured Materials, 2007-07-09, Alvor, Portugal).

*Milenkovic, S., A. J. Smith and A. W. Hassel:* Self-organised Metallic Nanostructures via Directional Solidification of Eutectics. (EUROMAT 2007, European Congress and Exhibition on Advanced Materials and Processes, 2007-09-10 to 2007-09-13, Nürnberg).

*Milenkovic, S., G. Frommeyer and A. Schneider:* Mechanical behaviour of the NiAl-W eutectic alloys. (EUROMAT 2007, European Congress and Exhibition on Advanced Materials and Processes, 2007-09-10 to 2007-09-13, Nürnberg).

*Milenkovic, S. and A. W. Hassel:* Metallic Nanostructures Self-organisation during Directional Solidification of Eutectics. (GDCh Wissenschaftsforum 2007, 2007-09-16 to 2007-09-19, Ulm).

*Moscicki, M., H. Pinto, C. Paulmann and A. R. Pyzalla:* An Automatic Procedure for Residual Stress Analysis in Individual Crystallites of a Polycrystalline Material. (Euromat 2007, 2007-09-10 to 2007-09-13, Nürnberg).

*Neelakantan, L., G. Eggeler and A. W. Hassel:* Electropolishing of NiTi - Insight into its mechanism. (58<sup>th</sup> Annual Meeting of the International Society of Electrochemistry, 2007-09-09 to 2007-09-14, Banff, Canada).

*Neelakantan, L., G. Eggeler and A. W. Hassel:* Electropolishing of NiTi and Surface Changes due to Phase Transformation. (GDCh Wissenschaftsforum 2007, 2007-09-16 to 2007-09-19, Ulm).

*Neelakantan, L., G. Eggeler and A. W. Hassel:* Electropolishing of NiTi - A mechanistic study. (212<sup>th</sup> ECS Meeting, 2007-10-07 to 2007-10-12, Washington (DC), USA).

*Neelakantan, L., K. Fushimi, G. Eggeler and A. W. Hassel:* Electropolishing and Electrochemical Micromachining of NiTi. (SMST 2007 (The International Conference on Shape Memory and Superelastic Technology), 2007-12-03 to 2007-12-05, Tsukuba, Japan).

*Neugebauer, J. (invited):* Ab initio based Multiscale Growth and Doping Simulations of Group-III Nitrides. (UK Nitrides Consortium Meeting, 2007-01-04 to 2007-01-05, Cambridge, UK).

*Neugebauer, J.* (invited): Ab initio basiertes Materialdesign auf dem Computer. (Hahn-Meitner-Institut Berlin, Colloquium, 2007-01-25, Berlin).

*Neugebauer, J.* (invited): Ab initio Thermodynamik in der Materialwissenschaft: Status und Perspektiven. (Fraunhofer Institut für Werkstoffmechanik Freiburg, Colloquium, 2007-03-16, Freiburg).

*Neugebauer, J.* (invited): Ab initio based growth simulations of group-III-nitrides. (Fifth International Conference on Solid State Crystals & Eighth Polish Conference on Crystal Growth, 2007-05-20 to 2007-05-25, Zakopane, Poland).

*Neugebauer, J.* (invited): Computer assisted design of metastable alloys. (Leibniz-Institut für Festkörper und Werkstofforschung, International Workshop, 2007-05-26, Dresden).

*Neugebauer, J.* (invited): Ab initio Determination of Thermodynamic Quantities: Status and Perspectives. (The first Sino-German Symposium on Computational Thermodynamics and Kinetics and their Applications to Solidification, 2007-05-31 to 2007-06-05, Changsha, China).

*Neugebauer, J.* (invited): Thermodynamic versus Kinetic Stabilization of Short and Long-Range Order in Nitride Based Alloys. (Multiscale approach to alloys: Advances and challenges, 2007-06-17 to 2007-06-19, Sigtuna, Sweden).

*Neugebauer, J.* (invited): First-principles calculations on CVD growth and doping in group-III-nitride semiconductors. (EuroCVD16 - Sixteenth European Conference on Chemical Vapor Deposition, 2007-09-16 to 2007-09-21, Den Haag, The Netherlands).

*Neugebauer, J.* (invited): Ab initio thermodynamics. (International Max-Planck Workshop Multiscale Materials Modeling of Condensed Matter, 2007-10-28 to 2007-11-02, Sant Feliu de Guixols, Spain).

*Neugebauer, J.* (invited): Ab initio thermodynamic and kinetics based on material design: present status and perspectives. (Seminar at the University of Oxford, Department of Materials, 2007-11-23, Oxford, UK).

*Nikolov, S., C. Sachs, H. Fabritius and D. Raabe:* Microstructure and micromechanics of hard biological tissues: From lobster cuticle to human bone. (Université Catholique de Louvain, Department of Applied Sciences, 2007-02-02, Louvain, Belgium).

*Nikolov, S., C. Sachs, A. Counts, H. Fabritius and D. Raabe:* Modeling of the Mechanical Behavior of Bone at Submicron Scale through Mean-Field Homogenization. (European Congress and Exhibition on Advanced Materials and Processes (EUROMAT 2007), 2007-09-10 to 2007-09-13, Nürnberg).

*Özcan, O., P. Thissen, A. T. Blumenau and G. Grundmeier:* Adsorption of organosilane molecules on polar ZnO (0001) surfaces. (ECASIA 2007, 12<sup>th</sup> European Conference on Applications of Surface and Interface Analysis, 2007-09-09 to 2007-09-14, Brussels-Flagey, Belgium).

*Özcan, Ö., P. Thissen, D. Diesing, A. T. Blumenau and G. Grundmeier:* A Monte Carlo - DFT Study: Adsorption of organosilanes on polar ZnO(0001) surfaces. (43<sup>th</sup> Symposium on Theoretical Chemistry, 2007-09-16 to 2007-09-20, Saarbrücken).

*Palm, M.* (invited): Innovative Fe-Al based materials. (15. Sitzung des DGM FA Intermetallische Phasen, 2007-01-10, Düsseldorf).

*Palm, M.* (invited): Strengthening mechanisms for Fe-Al-based alloys with increased creep resistance at high temperatures. (Charles University, 2007-08-26, Prague, Czech Republic)

*Paninski, M., A. Drevermann, G. J. Schmitz, M. Palm, F. Stein, M. Heilmaier, N. Engberding, H. Saage and D. Sturm:* Casting and properties of Al-rich Ti-Al alloys. (11<sup>th</sup> World Conference on Titanium, Ti-2007, 2007-03-03 to 2007-03-07, Kyoto, Japan).

*Petrov, M., L. Lympirakis and J. Neugebauer:* Nonlinear Elastic Effects in Group III-Nitrides. (Mid-term Review Meeting of PARSEM, 2007-02-06 to 2007-02-07, Berlin).

*Petrov, M., M. Friák, L. Lympirakis, J. Neugebauer and D. Raabe:* Hardness anisotropy of crystalline alpha-chitin: An ab-initio based conformational analysis. (Spring meeting of the German Physical Society (DPG), 2007-03-26 to 2007-03-30, Regensburg).

*Petrov, M., L. Lympirakis and J. Neugebauer:* Nonlinear Elastic Effects in Group III-Nitrides. (Spring meeting of the German Physical Society (DPG), 2007-03-26 to 2007-03-30, Regensburg).

*Petrov, M., L. Lympirakis, J. Neugebauer, R. Stefaniuk and P. Dłuzewski:* Nonlinear Elastic Effects in Group III-Nitrides: From ab-initio to Finite Element Calculation. (17<sup>th</sup> International Conference on Computer Methods in Mechanics, CMM-2007, 2007-06-19 to 2007-06-22, Spala, Poland).

*Petrov, M., M. Friák, L. Lympirakis and J. Neugebauer:* An ab-initio study of hardness anisotropy of crystalline alpha-chitin. (Euromat 2007, 2007-09-10 to 2007-09-13, Nürnberg).

*Petrov, M., M. Friák, L. Lympirakis, J. Neugebauer and D. Raabe:* An ab-initio study of hardness anisotropy of crystalline alpha-chitin. (International Max-Planck Workshop on Multiscale Modeling of Condensed Matter, 2007-10-28 to 2007-11-02, Sant Feliu de Guixols, Spain).



- Petrov, M., M. Friák, L. Lymperekis, D. Raabe and J. Neugebauer:* Ab initio based multiscale description of biological and biocompatible materials. (Colloquium lecture at Max Planck Institute for Colloids and Interfaces, 2007-11-22, Golm).
- Pinto, H.* (invited): Stress & texture studies. (5<sup>th</sup> European Winter School (NESY 2007) Research with Neutron and Synchrotron Radiation, 2007-03-05 to 2007-03-09, Plannersalm, Donnersbach, Austria).
- Pinto, H., P. A. Silva, A. Kostka, H. F. Chladil, H. Clemens and A. R. Pyzalla:* Microstructure and Residual Stress Formation in Oxide Layers Grown on the Gamma-Ti-45Al-5Nb Alloy. (Euromat 2007, 2007-09-10 to 2007-09-13, Nürnberg).
- Pinto, H., M. Petrov, M. Friák, L. Lymperekis, H. Fabritius, J. Neugebauer, D. Raabe and A. R. Pyzalla:* Residual Stresses in the Exoskeleton of a Homarus americanus Lobster. (MECASSENS IV, 2007-09-24 to 2007-09-26, Vienna, Austria).
- Pinto, H., M. Petrov, M. Friák, L. Lymperekis, H. Fabritius, J. Neugebauer, D. Raabe and A. R. Pyzalla:* Eigenspannungen am Hummerexoskelett. (Sitzung des FA 13 Eigenspannungen am AWT, 2007-11-07, Leibniz-Institut für Festkörper- und Werkstoffforschung Dresden (IFW), Dresden).
- Pinto, H., C. Juricic, C. Genzel and A. R. Pyzalla:* Effect of substrate microstructure on phase, texture and internal stress evolution in iron oxide layers grown at 650 °C. (Zeit- und temperaturaufgelöste Röntgen-Pulver-Diffraktometrie VIII, Fraunhofer ICT, 2007-11-08 to 2007-11-09, Pfinztal).
- Ponge, D., Z. Huang, F. Roters and D. Raabe:* Grundlagen der gefügeabhängigen Modellierung von Warmumformprozessen. (VDI Tagung Massivumformung, 2007-09-27, Leonberg).
- Ponge, D.* (invited): The formation of ultrafine grained microstructure in a plain C-Mn steel. (International Symposium of Ultrafine Grained Steels ISUGS-2007, 2007-10-24 to 2007-10-26, Kitakyushu, Japan).
- Posner, R., K. Wapner, R. Vlasak and G. Grundmeier:* Combined Spectroscopic and Electrochemical Studies of Water and Ion Transport at Polymer/Oxide/Metal Interfaces. (ECASIA 2007, 12<sup>th</sup> European Conference on Applications of Surface and Interface Analysis, 2007-09-09 to 2007-09-14, Brussels-Flagey, Belgium).
- Prymak, O., M. Palm and F. Stein:* Phase equilibria in the Nb-Cr-Al system. (Workshop: The Nature of Laves Phases VIII, 2007-01-11, Duesseldorf).
- Prymak, O., F. Stein, G. Frommeyer and D. Raabe:* Laves phases in the ternary Nb-Cr-Al system: Phase equilibria at 1150, 1300 and 1450 °C. (EUROMAT 2007, 2007-09-10 to 2007-09-13, Nürnberg).
- Prymak, O., F. Stein, G. Frommeyer and D. Raabe:* Phase equilibria in the Nb-Cr-Al system at 1150, 1300 and 1450 °C. (Workshop: The Nature of Laves Phases IX, 2007-09-20 to 2007-09-21, Stuttgart).
- Pyzalla, A. R.* (invited): Stahlforschung am MPI. (Seminar des Instituts für Werkstoffe, 2007-01-08, Universität Kassel, Kassel).
- Pyzalla, A. R.* (invited): Neue Möglichkeiten zur Werkstoff- und Schädigungscharakterisierung mit Synchrotronstrahlung. (BAM Kolloquium, 2007-01-16, Berlin).
- Pyzalla, A. R.* (invited): Werkstoffdiagnostik und Technologie der Stähle am Max-Planck-Institut für Eisenforschung. (MPG Senatssitzung, 2007-03-22, Munich).
- Pyzalla, A. R., A. Isaak, F. Sket, K. Dzieciol and W. Reimers* (invited): In-situ Untersuchung der Entwicklung der Kriechschädigung in metallischen Werkstoffen. (53. Metallkunde-Kolloquium, 2007-04-16 to 2007-04-18, Lech, Austria).
- Pyzalla, A. R.* (invited): Werkstoffdiagnostik. (Michelkreis, 2007-07-05 to 2007-07-06, Luxembourg, Luxembourg).
- Pyzalla, A. R., A. Isaac, F. Sket, K. Dzieciol and G. Sauthoff* (invited): Investigation of creep damage evolution in alloys and composites using synchrotron radiation. (2007 Denver X-ray Conference, 2007-07-30 to 2007-08-03, Colorado, USA).
- Pyzalla, A. R. and A. Borbély* (invited): Tomography investigations of engineering materials. (1<sup>st</sup> GKSS/DESY Workshop for Mikro & Nano Tomographie, 2007-09-14, Hamburg).
- Pyzalla, A. R.* (invited): Residual stresses in engineering materials. (VI-PNAM Autumn School: Application of Neutrons and Synchrotron Radiation in Engineering Materials Science, 2007-09-17 to 2007-09-21, Hamburg).
- Pyzalla, A. R.* (invited): Förderung des wissenschaftlichen Nachwuchses in der MPG. (GAIN-Jahrestagung, 2007-09-21 to 2007-09-23, San Francisco, USA).
- Pyzalla, A. R.* (invited): Tomography combined with diffraction. (2<sup>nd</sup> GKSS/DESY Workshop High Energy Materials Science at PETRA III, 2007-11-02, Hamburg).
- Pyzalla, A. R., A. Isaac, F. Sket, K. Dzieciol, G. Sauthoff and A. Borbély* (invited): In-situ Characterisation of Creep Damage Evolution in Metallic Materials using Synchrotron Tomography. (Symposium: Microstructural Characterisation down to the Atomic Scale, 2007-12-04 to 2007-12-05, Leoben, Austria).
- Raabe, D.* (invited): 3D analysis of nanoindentation in metallic single crystals. (Sixth GAMM Seminar on Microstructures, 2007-01-12, Berlin).



*Raabe, D.* (invited): Simulation methods in materials science. (Lecture at Akademie der Wissenschaften und der Literatur, Symposium: Digitale Modellierung, Simulation und Visualisierung, 2007-03-09, Mainz).

*Raabe, D.* (invited): Crystal Mechanics of Metals and Biological Matter. (Colloquium lecture at Max Planck Institute for Colloids and Interfaces, 2007-03-20, Golm).

*Raabe, D., H. Fabritius, L. Raue, H. Klein, C. Sachs, A. Al-Sawalmih and S.-B. Yu* (invited): Textures of biological matter. (Deutsche Gesellschaft für Materialkunde e.V., Fachausschuss Texturen, 2007-03-23, Aachen).

*Raabe, D.* (invited): Multiscale crystal mechanics at small and at large scales. (Imperial College, 2007-04-17, London, UK).

*Raabe, D., N. Zaafarani, F. Roters and S. Zaefferer* (invited): 3D studies on orientation patterning below nanoindents in Cu single crystals using 3D EBSD and crystal plasticity finite element simulations. (3<sup>rd</sup> International Indentation Workshop, 2007-07-17, Cavendish Laboratory, Cambridge, UK).

*Raabe, D.* (invited): Crystal mechanics of metals and biomaterials at small and at large scales. (Ecole Centrale, 2007-08-17, Paris, France).

*Raabe, D.* (Keynote Lecture): Basic Concepts of Internal Variable Modeling (VDI Massivumformtagung, 2007-09-27, Leonberg).

*Raabe, D.* (Plenary Lecture): New approaches in multiscale modeling. (WING Conference in BMBF, 2007-10-24, Berlin).

*Raabe, D., F. Roters, P. Eisenlohr, S. Zaefferer, N. Zaafarani, E. Demir, A. Ma and D. Ma* (invited): Understanding the Elasto-Plasticity of Crystals. (MMM- Max-Planck Multiscale Simulation Conference, 2007-10-28 to 2007-11-02, Sant Feliu de Guixols, Spain).

*Raabe, D., F. Roters, D. Ma, S. Zaefferer, M. Friák and N. Zaafarani* (invited): Orientation patterning below indents and bottom-up mechanical design by using quantum mechanics. (Symposium Multiscale Plasticity of Crystalline Materials of the International Union of Theoretical and Applied Mechanics (IUTAM), 2007-11-06, Eindhoven, The Netherlands).

*Rablbauer, R., K. Dönecke, A. W. Hassel and G. Frommeyer:* Mechanical properties and corrosion behaviour of ferritic stainless Al Cr steels. (EUROMAT 2007, European Congress and Exhibition on Advanced Materials and Processes, 2007-09-10 to 2007-09-13, Nürnberg).

*Rablbauer, R. and G. Frommeyer:* Mechanical properties and deformation mechanism of high-strength lightweight MnAlC TRIPLEX steels. (EUROMAT 2007, 2007-09-10 to 2007-09-13, Nürnberg).

*Rasp, W. and D. Filatov:* Verhalten des in der Sandwichprobe eingeschlossenen Zunders im Walzspalt beim Warmwalzen. (Vortrag VDEh-Walzwerksausschuss, UA Grobblech, 2007, Linz, Austria).

*Rinke, P., M. Scheffler, A. Qteish and J. Neugebauer:* Band gap and band parameters of InN from quasiparticle energy calculations based on exact-exchange density-functional theory. (Spring meeting of the German Physical Society (DPG), 2007-03-26 to 2007-03-30, Regensburg).

*Rohwerder, M.* (invited): Intelligent corrosion protection by conducting polymers without drawbacks. (European Coatings Conference, 2007-06-14 to 2007-06-15, Berlin).

*Rohwerder, M.:* Kelvin Probe, Work Function, Absolute Potentials. (Workshop on Converging Theoretical and Experimental Approaches to Corrosion Science, 2007-11-07, MPIE, Düsseldorf).

*Rohwerder, M.:* Inherent Delamination Protection by Novel Zinc Alloys. (GALVATECH '07, 7<sup>th</sup> International Conference on Zinc and Zinc Alloy Coated Steel Sheet, 2007-11-18 to 2007-11-22, Osaka, Japan).

*Romano, P., S. Zaefferer:* Identification of Bainite in Al-TRIP Steels using orientation microscopy. (14<sup>th</sup> Conference on Electron Backscatter Diffraction, 2007-03-27, New Lanark, UK).

*Roters, F.* (invited): Strain Gradient Plasticity with Internal Interfaces. (Workshop on Small Scale Plasticity, 2007-09-04 to 2007-09-08, Braunwald, Switzerland).

*Sachs, C., H. Fabritius, S. Nikolov and D. Raabe:* Influence of structural principles on the mechanics and efficiency of different biological materials using lobster cuticle as a model material. (Spring Meeting of the German Physical Society (DPG), 2007-03-26, Regensburg).

*Sachs, C., H. Fabritius, F. Pitsch and D. Raabe:* Nanoindentation as tool to investigate micro-mechanical properties in the hierarchical structure of biological materials. (MRS Fall Meeting, 2007-11-28, Boston, USA).

*Sander, B., D. Ma, M. Friák, F. Roters, N. Zaafarani, S. Zaefferer, J. Neugebauer and D. Raabe:* 1) Design of new Ti-based biomaterials by using ab-initio simulations, FEM and experiments 2) Detailed analysis of an indent. (AICES – 2007 MIT conference, RWTH Aachen, Aachen).

*Sanders, D., S. Kaufhold, R. Dohrmann and A. W. Hassel:* Corrosion of steel and copper in clay rich soils. (The European Corrosion Congress, 2007-09-09 to 2007-09-13, Freiburg).



*Scharnberg, M., V. Zaporozhchenko, R. Adelung, S. Milenkovic and A. W. Hassel:* Reflection of water jets on biological and bio-inspired artificial surfaces. (Spring meeting of the German Physical Society (DPG), 2007-03-26 to 2007-03-30, Regensburg).

*Schölzel, M., M. Scharnberg, R. Adelung and A. W. Hassel:* Kontaktwinkelmessungen an funktionalisierten, nanostrukturierten Metalloberflächen. (GDCh Wissenschaftsforum 2007, 2007-09-16 to 2007-09-19, Ulm).

*Schuster, J. C., S. Ackerbauer, N. Krendelsberger, S. Liu, M. Naka, M. Palm, F. Stein and F. Weitzer:* THALU + silicon: Recent experimental ternary phase diagram data. (Thermodynamics of Alloyed Aluminides, THALU, 2007-10-21 to 2007-10-24, Interlaken, Switzerland).

*Sket, F., A. Isaac, K. Dzieciol and A. R. Pyzalla:* Caracterizacion in-situ en 3D de danos durante creep usando XMT. (Jornadas de Investigadores Iberoamericanos en Ciencia de Materiales, 2007-05-23 to 2007-05-26, Alicante, Spain).

*Sket, F., A. Isaac, K. Dzieciol, A. Borbély, G. Sauthoff and A. R. Pyzalla:* Quantitative assessment of inhomogeneous creep cavity distribution. (E-MRS Fall Meeting 2007, 2007-09-16 to 2007-09-22, Warsaw, Poland).

*Sket, F., A. Isaac, K. Dzieciol and A. R. Pyzalla (invited):* Caracterizacion in-situ en 3D de Danos durante creep usando XMT. (Seminario de Ciencias de Mateariales, 2007-11-08 to 2007-11-10, Huelva, Spain).

*Smith, A. J., B. Bello-Rodriguez, S. Milenkovic and A. W. Hassel:* Overview of Nanoscopic Morphologies Accessible Through Electrochemical Processing of Directionally Solidified Eutectic Alloys. (92. AGEF-Seminar DFG-SPP 1165: Elektrochemische Aspekte von Nanodrähten und Nanoröhren, 2007-04-18, Düsseldorf).

*Smith, A. J., S. Milenkovic and A. W. Hassel:* Elektrochemische Aspekte von Nanodrähten und Nanoröhren. (92. AGEF-Seminar DFG-SPP 1165: Elektrochemische Aspekte von Nanodrähten und Nanoröhren, 2007-04-18, Düsseldorf).

*Smith, A. J., S. Milenkovic and A. W. Hassel:* Initial steps in handling single nanowires. (92. AGEF-Seminar DFG-SPP 1165: Elektrochemische Aspekte von Nanodrähten und Nanoröhren, 2007-04-18, Düsseldorf).

*Smith, A. J., M. Stratmann and A. W. Hassel:* Investigation of Erosion - Corrosion Phenomena with the Help of Single Impact Impingement Studies. (2007 Spring Meeting of the Japan Society for Corrosion Engineering Materials and Environments, 2007-05-09 to 2007-05-11, Tokyo, Japan).

*Smith, A. J., A. W. Hassel and S. Milenkovic:* Selective Electrodeposition of Directionally Solidified Eutectic Alloys for 'Mass Production' of Metallic Nanowires with Extreme Aspect Ratios. (58<sup>th</sup> Annual Meeting of the International Society of Electrochemistry, 2007-09-09 to 2007-09-14, Banff, Canada).

*Stein, F., G. Frommeyer and A. Schneider:* Microstructure, atomic ordering, surface tension and viscosity of  $\mu\text{g}$ -processed iron-aluminium-silicon alloys with up to 6.5 wt.% Al+Si. (Meeting: TEMPUS Parabolic Flight 2006, 2007-03-03, German Aerospace Center (DLR), Bonn).

*Stein, F., L. Siggelkow, G. Kreiner and M. Palm:* Unusual mechanical behaviour of the intermetallic phase  $\text{Nb}_2\text{Co}_7$ . (Workshop: The Nature of Laves Phases IX, 2007-09-20 to 2007-09-21, Stuttgart).

*Stratmann, M. (invited):* In-situ SKP study of the diffusion of ions along buried interfaces. ECS Meeting 2007, 2007-10-08, Washington (DC), USA

*Sturm, D., H. Saage, M. Heilmaier, M. Paninski, G. J. Schmitz, M. Palm, F. Stein, A. Drevermann and N. Engberding:* Mechanical properties of centrifugally cast Al-rich TiAl alloys. (EUROMAT 2007, 2007-09-10 to 2007-09-13, Nürnberg).

*Swaminathan, S. and M. Spiegel:* Effect of alloy composition on the selective oxidation of ternary Fe-Si-Cr, Fe-Mn-Cr model alloys. (ECASIA 2007, 12<sup>th</sup> European Conference on Applications of Surface and Interface Analysis, 2007-09-09, Brussels-Flagey, Belgium).

*Swaminathan, S. and M. Rohwerder:* Hot-dip galvanizing simulation of model alloys and industrial steel grades: Correlation between surface chemistry and wettability. (GALVATECH '07, 7<sup>th</sup> International Conference on Zinc and Zinc Alloy Coated Steel Sheet, 2007-11-18 to 2007-11-22, Osaka, Japan).

*Theisen, W. and S. Weber:* Supersolidus-Flüssigphasensintern von Werkzeugstählen und Fe-Basis-MMC. (Hagener Symposium Pulvermetallurgie 2007, 2007-11-29 to 2007-11-30, Hagen).

*Thissen, P., M. Giza and G. Grundmeier:* Adsorptionskinetik von Organophosphonsäure auf plasmamodifizierten Aluminiumoberflächen. (13. Bundesdeutsche Fachtagung für Plasmatechnologie, 2007-03-05 to 2007-03-07, Bochum).

*Thissen, P., M. Giza and G. Grundmeier:* Adsorptions kinetics of organophosphonic acids on plasma modified aluminium surfaces. (ECASIA 07, 12<sup>th</sup> European Conference on Applications of Surface and Interface Analysis, 2007-09-09 to 2007-09-14, Brussels-Flagey, Belgium).

*Thissen, P., Ö. Özcan, D. Diesing and G. Grundmeier:* Monte Carlo Simulation of Temperature Programmed Desorption Including Binding Energies and Frequency Factors Derived by DFT Calculations. (43<sup>th</sup> Symposium on Theoretical Chemistry, 2007-09-16 to 2007-09-20, Saarbrücken).

*Thissen, P., Ö. Özcan, E. Torres, D. Diesing and G. Grundmeier:* Combining Monte Carlo Kinetics and Density Functional Theory to simulate Temperature Programmed Desorption. (American Vacuum Society 54<sup>th</sup> International Symposium, 2007-10-14 to 2007-10-19, Seattle, USA).

*Titz, T., M. Giza and G. Grundmeier:* Structure and properties of ultra-thin SiO<sub>2</sub> plasma polymer films at polymer/metal interfaces. (13. Fachtagung Plasmatechnologie (PT13), 2007-03-05 to 2007-03-07, Bochum).

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*Zaefferer, S. (invited):* 3D orientation microscopy in a FIB-SEM: A new dimension of microstructure characterisation, (presentation at the scientific advisory board, 2007-02-27, MPIE, Düsseldorf).

*Zaefferer, S. (invited):* 3D orientation microscopy in a FIB-SEM: A new dimension of microstructure characterisation, (Research Metallica Chair 2007, 2007-03-07, KU Leuven, Leuven, The Netherlands).

*Zaefferer, S. (poster and talk):* Some ideas on the formation mechanisms and intensity of electron backscatter diffraction patterns, (14<sup>th</sup> Conference on Electron Backscatter Diffraction, 2007-03-27, New Lanark, UK).

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*Zaefferer, S. and P. Romano:* Identification and quantification of microstructural constituents in low-alloyed TRIP steels by simultaneous EBSD and EDS measurements. (Microscopy and Microanalysis 2007 Meeting, 2007-08-05 to 2007-08-09, Fort Lauderdale, USA).

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*Zaefferer, S. (invited):* 3D orientation microscopy by Combined FIB-serial sectioning EBSD-based orientation microscopy: principles and applications. (FEMMS 2007, 2007-09-25, Sonoma, USA).

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*Abu-Farsakh, H. and J. Neugebauer:* Solubility and surface kinetics of N in diluted nitrides. (Computational Materials Science Workshop, 2008-08-25 to 2008-08-28, Schloss Eberburg, Bad Münster am Stein-Eberburg).

*Al-Sawalmih, A., H. Fabritius, S. Yi, C. Li, S. Siegel, D. Raabe and O. Paris:* Investigation of the Orientation Relationship Between  $\alpha$ -Chitin and Calcite in Crustacean Cuticle Using Microbeam Synchrotron X-ray Diffraction. (15<sup>th</sup> International Conference on the Texture of Materials (ICOTOM 15), 2008-06-01 to 2008-06-06, Carnegie Mellon University Center, Pittsburgh, USA).

*Amberger, D., P. Eisenlohr and M. Göken:* Microstructural evolution during creep of Ca-containing AZ91. (Creep 2008, 2008-05-04 to 2008-05-09, Bad Berneck).

*Ankah, G. N., F. U. Renner and M. Rohwerder:* Fundamental investigations of the corrosion of binary alloys. (59<sup>th</sup> Annual Meeting of the International Society of Electrochemistry, 2008-09-07 to 2008-09-12, Seville, Spain).

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- Balasundaram, K., Y. Cao and D. Raabe:* Nanomechanics characterization of polymer. (MPIE-Interdepartmental tutorial day (s), 2008-02-13, MPIE, Düsseldorf).
- Balasundaram, K., Y. Cao and D. Raabe:* Nanomechanics characterization of softmatter using nanoindentation. (11<sup>th</sup> GLADD Meeting, 2008-04-25, TU Gent, Gent, Belgium).
- Balasundaram, K., Y. Cao and D. Raabe:* Investigating the Applicability of the Oliver & Pharr Method to the Nano-Mechanical Characterization of Soft Matter. (1<sup>st</sup> International Conference from Nanoparticles and Nanomaterials to Nanodevices and Nanosystems, 2008-06-16 to 2008-06-18, Halkidiki, Greece).
- Balasundaram, K., Y. Cao and D. Raabe:* Nano-mechanical Characterization of Soft Matter. (Materials science Day, 2008-07-17, Mechanical Engineering Department at Ruhr-Universität Bochum, Bochum).
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- Barbatti, C., H. Pinto and A. R. Pyzalla:* Defect and Stress Analyses in Novel Fe-Mn-C Steels by X-Ray Diffraction. (MSE08 Materials Science and Engineering, 2008-09-01 to 2008-09-04, Nürnberg).
- Biedermann, P. U.:* Electrochemical oxygen reduction mechanism in aqueous solution. (3<sup>rd</sup> Computational Materials Science Workshop, 2008-08-25 to 2008-08-28, Schloss Eberburg, Bad Münster am Stein-Eberburg).
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- Blum, W. and P. Eisenlohr:* Dislocation mechanics of creep. (Creep 2008, 2008-05-04 to 2008-05-09, Bad Berneck).
- Bobrowski, A., K. Brokmeier, G. Frommeyer, A. Maier and R. Rablbauer:* Metallphysikalische Verformungsmechanismen hoch manganhaltiger austenitischer Stähle. (Materialographie 2008 - 42. Metallographie-Tagung, 2008-09-17 to 2008-09-19, Jena).
- Borbély, A., C. Maurice and J. H. Driver:* Rotation-axis analysis of deformation induced dislocation boundaries. (TMS 2008 annual meeting, Hael Mughrabi Honorary Symposium: Plasticity, Failure and Fatigue in Structural Materials, 2008-03-09 to 2008-03-13, New Orleans, USA).
- Borbély, A.:* Quo vadis XLP? (Ungár Tamás Honorary Symposium, X-ray Line Profile Analysis, 2008-05-19 to 2008-05-20, Eötvös Loránd University, Budapest, Hungary).
- Borbély, A.:* 3D experiment data using diffraction microscopy. (ICOTOM 15, 2008-06-01 to 2008-06-06, Carnegie Mellon University Center, Pittsburg, USA).
- Borbély, A., K. Dzieciol, A. Isaac, F. Sket and A. R. Pyzalla (invited):* Application of microtomography to engineering materials. (Workshop on X-Ray Micro Imaging of Materials, Devices and Organisms, 2008-10-22 to 2008-10-24, Dresden).
- Borissov, D., F. U. Renner and M. Rohwerder:* Zn-Mg-Al Alloy Electrodeposition from Ionic Liquids. (59<sup>th</sup> Annual Meeting of the International Society of Electrochemistry, 2008-09-07 to 2008-09-12, Seville, Spain).
- Borodin, S. and M. Rohwerder:* Preparation of model single crystalline aluminium oxide films suitable for scanning tunnelling microscopy. (Spring meeting of the German Physical Society (DPG), 2008-02-25 to 2008-02-29, Berlin).
- Borodin, S. and M. Rohwerder (invited):* Fitting of XPS metallic signals with an asymmetrical part. (PHI User Meeting, 2008-04-22, Ismaning).
- Borodin, S. and M. Rohwerder:* Growth of high quality crystalline aluminium oxide films on Nb(110)/sapphire(11-20). (14<sup>th</sup> International Conference on Solid Films and Surfaces (ICSFS), 2008-06-29 to 2008-07-04, Dublin, Ireland).
- Bouquerel, J., K. Verbeken, J. Van Slycken, P. Verleysen and Y. Houbaert:* Physically based modeling of the mechanical behavior of TRIP steels. (11<sup>th</sup> ESAFORM2008 conference on material forming, 2008-04-23 to 2008-04-25, Lyon, France).
- Bouquerel, J., K. Verbeken, J. Van Slycken, P. Verleysen and Y. Houbaert:* Simulation of the static mechanical behaviour of trip steels by micro-mechanical modelling. (New developments on Metallurgy and applications of high strength steels, 2008-05 26 to 2008-05-28, Buenos Aires, Argentina).
- Brahme, A.:* Brief Introduction to Cellular Automaton and Monte Carlo Method. (MPIE Inter-Departmental Tutorial day(s) 2008, 2008-04-16, MPIE, Düsseldorf).

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*Brito, P., H. Pinto, M. Spiegel, M. Klaus, C. Genzel and A. R. Pyzalla:* Phase composition and internal stress development during the oxidation of iron aluminides. (ICRS-8, 2008-08-06 to 2008-08-08, Denver, USA).

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*Bruder, K., A. W. Hassel and D. Diesing:* MIM Characterisation of thin tantalum oxide films. (Spring meeting of the German Physical Society (DPG), 2008-02-25 to 2008-02-29, Berlin).

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*Frommeyer, G.* (invited): Entwicklung, Mikrostrukturen und Eigenschaften hochfester und supraduktiler Leichtbaustähle aus Fe-Mn-Al-Si-(C) für zukünftige Designkonzepte im Fahrzeugbau. (Forum für Metallurgie und Werkstofftechnik 2008, 2008-05-19 to 2008-05-20, Leoben, Austria).

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*Fujita, N.*: Atomistic modelling of nitrogen related defects in silicon. (5<sup>th</sup> Forum on the Science and Technology of Silicon Materials, 2008-11-12 to 2008-11-14, Niigata, Japan).

*Garcia, L. F., J. Garcia, A. Kostka, S. Weber and A. Lammer*: Wear Behaviour of Cooper-Iron-Cobalt Diamond Tool Bonding Matrices Reinforced with Hardmetal Granulates. (9<sup>th</sup> International Conference on the Science of Hard Materials, 2008-03-10 to 2008-03-14, Montego Bay, Jamaica).

*Garcia, J.* (invited): Advanced Engineering Materials Network - Managing an ALFA II Project. (Symposium ALFA III, Deutscher Akademischer Austauschdienst (DAAD), 2008-04-10, Bonn).

*Garcia, J. and S. Weber* (invited): Analytics and alloy development of creep resistant steels at the Material Diagnostic and Steel Technology Department of MPIE. (COST 536 Meeting, 2008-04-11, Bergamo, Italy).

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*Grundmeier, G.*: Tailored thin plasma polymer and plasma polymer/metal nanocomposite films on metals - From corrosion protection to multifunctional properties. (1<sup>st</sup> International Conference on Functional Nanocoatings, 2008-03-30 to 2008-04-02, Budapest, Hungary).

*Grundmeier, G. and R. Posner* (invited): Structure and stability of polymer/oxide/metal interfaces - Bridging the gap between interface analysis and advanced surface technologies. (CoSI 2008, Coating Science International conference, 2008-06-23 to 2008-06-27, Noordwijk, The Netherlands).

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*Hassel, A. W. and S. Milenkovic*: Single crystalline metal nanowires with high aspect ratio. (Spring meeting of the German Physical Society, 2008-02-25 to 2008-02-29, Berlin).

*Hassel, A. W.* (invited): Arrays of Iso-Oriented Metal Nanowires. (ICYS Final Workshop, 2008-03-10 to 2008-03-13, Tsukuba, Japan).

*Hassel, A. W.*: Nanodrähte und Nanodrahtarrays und abgeleitete Strukturen durch chemische Prozessierung gerichtet erstarrter Eutektika. (Chemisches Kolloquium der Fakultät für Chemie und Biochemie, 2008-04-10, Ruhr-Universität Bochum, Bochum).

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*Hassel, A. W. and A. J. Smith*: Mechanical Testing and Manipulation of Metal Nano Wires. (Kleindiek User Meeting, 2008-05-01 to 2008-05-03, Reutlingen).

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- Hassel, A. W.* (invited): Metal nanowires – electrochemical release, modification and applications. (24<sup>th</sup> One-Day-Symposium, Electrochemical Materials Processing, 2008-05-07, Villigen, Schweiz).
- Hassel, A. W., M. Stratmann, K. A. Lill and A. I. Mardare* (invited): The scanning droplet cell in corrosion research. (8<sup>th</sup> Israel Conference on Corrosion, 2008-05-21, Haifa, Israel).
- Hassel, A. W.* (invited): Nanostructuring of alloy surfaces. (Colloquium des Sonderforschungsbereichs 569, 2008-06-17, Ulm).
- Hassel, A. W.* (invited): Tailoring of Nanostructured Alloys by Anodisation. (International Symposium on Anodizing Science and Technology 2008, 2008-07-23 to 2008-07-25, Rusutsu, Japan).
- Hassel, A. W., K. A. Lill, A. I. Mardare and M. Stratmann* (invited): New Approaches in Materials Science with the Scanning Droplet Cell. (Seminarvortrag an der Universität von Hokkaido, 2008-07-28 Sapporo, Japan).
- Hassel, A. W.* (invited): Progress in the Electrochemical Processing of Directionally Solidified Eutectics. (7<sup>th</sup> International Symposium on Electrochemical Micro- and Nanosystems, 2008-09-07 to 2008-09-12, Ein-Gedi, Israel).
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- Hickel, T., B. Grabowski, F. Körmann, A. Dick and J. Neugebauer*: Ab initio determination of free energies and phase diagrams. (German-Korean miniworkshop, 2008-01-31, MPIE, Düsseldorf).
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- Hickel, T., M. Uijtewaal, B. Grabowski and J. Neugebauer*: First principles determination of phase transitions: The (pre)martensitic transition in Ni<sub>2</sub>(MnGa). (UCSB-MPG Workshop on Inorganic Materials for Energy Conversion, Storage and Conservation, 2008-02-19 to 2008-02-22, UCLA Lake Arrowhead Conference Center, Lake Arrowhead, USA).
- Hickel, T., M. Uijtewaal and J. Neugebauer*: Ab initio Investigations of Temperature Dependent Effects in MSM Alloys. (Antragskolloquium SPP1239, 2008-03-12, Dresden).
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- Hickel, T., M. Uijtewaal, B. Grabowski and J. Neugebauer* (invited): First principles determination of phase transitions in magnetic shape memory alloys. (XXI Congress of the International Union of Crystallography, 2008-08-23 to 2008-08-31, Osaka, Japan).
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- Hild, S.*: Isopoda: Crustacean models to study microstructure and properties of biological composite materials. 2008-01-24, MPIE, Düsseldorf
- Hild, S., O. Marti and A. Ziegler*: Isopod cuticle: A model system to study the influence of the structure and the chemical composition on the mechanics of biological composite materials. (Spring meeting of the German Physical Society (DPG), 2008-02-24 to 2008-02-29, Berlin).
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- Hild, S.*: Allocation of organic and inorganic components in isopoda. (Scanning Probe & Raman Microscopy Workshop, 2008-03-04, Cluj-Napoca, Romania).
- Hild, S. and A. Ziegler*: The isopod cuticle: A model to study formation and function of amorphous calcium carbonate in calcified tissues. (European Geosciences Union General Assembly, 2008-04-13 to 2008-04-18, Vienna, Austria).
- Hild, S.*: Isopod cuticle: A model for the characterization of the structure and the chemical composition of biocomposite materials. (BASF, 2008-06-10, Ludwigshafen).

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*Isaac, A., F. Sket, K. Dzieciol, G. Sauthoff, A. Borbély and A. R. Pyzalla:* In-situ investigation of creep damage using synchrotron X-ray microtomography. (VIII Simpósio Mecânica Computacional, SIMMEC 2008, 2008-07-25 to 2008-07-27, Belo Horizonte, Brazil).

*Ishikawa, S., M. Takeyama, S. Zaeferrer, M. Palm and F. Stein:* Phase equilibria and phase transformations of Laves phases in the Cr-Ti binary system. (JIM Annual Spring Meeting 2008, 2008-03-26 to 2008-03-28, Tokyo, Japan).

*Ismer, L., T. Hickel and J. Neugebauer:* First principles study of Hydrogen in Manganese-rich austenitic steels. (Spring meeting of the German Physical Society (DPG), 2008-02-25 to 2008-02-29, Berlin).

*Ismer, L., M. Friák, T. Hickel and J. Neugebauer:* Effect of interstitial carbon on the magnetic structure of fcc iron: Towards an ab-initio simulation of austenitic steels. (International Workshop on Ab initio Description of Iron and Steel (ADIS2008), 2008-06-15 to 2008-06-20, Schloss Ringberg, Kreuth).

*Ismer, L., J. Ireta and J. Neugebauer:* First principles study of vibrational and thermodynamic properties of the secondary structure of proteins. (Computational Materials Science Workshop, 2008-08-25 to 2008-08-28, Schloss Eberburg, Bad Münster am Stein-Eberburg).

*Itani, H., P. Keil and G. Grundmeier:* Silver Nanoparticles Embedded in LBL Deposited Polyelectrolyte Thin Films: Investigation of the Formation and Composition of the Silver Nanoparticles. (Advanced Processing for Novel Functional Materials APNFM, 2008-01-23 to 2008-01-25, Dresden).

*Itani, H., P. Keil and G. Grundmeier:* Silver Nanoparticles Embedded in Layer by Layer Polyelectrolyte Thin Films: Investigation of the Formation and Composition of Silver Nanoparticles. (First International Conference on FUNCTIONAL NANOCOATING, 2008-03-30 to 2008-04-02, Budapest, Hungary).

*Itani, H., P. Keil and G. Grundmeier:* Silver Nanoparticles Embedded in Layer by Layer Polyelectrolyte Thin Films: Investigation of the Formation and Composition of Silver Nanoparticles. (Polyelectrolytes 2008, 2008-07-19 to 2008-07-20, Coimbra, Portugal).

*Itani, H., P. Keil and G. Grundmeier:* Analytical Studies of the Formation of Silver Nanoparticles in Layer by Layer Deposited Polyelectrolyte Films. (IMPRS for Surface and Interface Engineering in Advanced Materials, 2008-08-12 to 2008-08-15, Schloss Gnadenenthal, Kleve).

*Khan, T. R., D. de la Fuente and M. Rohwerder:* Electrolytic co-deposition of SiO<sub>2</sub> nanoparticles with zinc for improvement of corrosion protection. (59<sup>th</sup> Annual Meeting of the International Society of Electrochemistry, 2008-09-07, Seville, Spain).

*Khorashadizadeh, A.:* Three-dimensional tomographic EBSD measurements of the crystal topology in heavily deformed ultra fine grained Cu-0.17wt%Zr obtained from ECAP. (Spring meeting of the German Physical Society (DPG), 2008-02-25 to 2008-02-29, Berlin).

*Kim, O., M. Friák and J. Neugebauer:* Ab initio analysis of the carbon solubility limits in various iron phases. (Spring meeting of the German Physical Society (DPG), 2008-02-25 to 2008-02-29, Berlin).

*Körmann, F., A. Dick, B. Grabowski, T. Hickel and J. Neugebauer:* Importance of magnetism for the thermal expansion of transition metals: An ab initio study. (Spring meeting of the German Physical Society (DPG), 2008-02-25 to 2008-02-29, Berlin).

*Körmann, F., A. Dick, B. Grabowski, T. Hickel and J. Neugebauer:* The free energy of bcc iron: Integrated ab initio derivation of vibrational, electronic and magnetic contributions. (International Workshop on Ab initio Description of Iron and Steel (ADIS2008), 2008-06-15 to 2008-06-20, Schloss Ringberg, Kreuth).

*Körmann, F., A. Dick, B. Grabowski, T. Hickel and J. Neugebauer:* The free energy of bcc iron: Integrated ab initio derivation of vibrational, electronic and magnetic contributions. (Computational Materials Science Workshop, 2008-08-25 to 2008-08-28, Schloss Eberburg, Bad Münster am Stein-Eberburg).

*Körmann, F., A. Dick, B. Grabowski, T. Hickel and J. Neugebauer:* Importance of magnetism for the thermodynamics of bcc iron: An ab initio study. (Summer School: Nanomagnetism and Spintronics, 2008-09-08 to 2008-09-17, Prague, Czech Republic).

*Kostka, A.:* EBSD Microstructure and Material Flow Characterization of an AA6181-T4 Aluminium Alloy to DP600 High Steel Friction Stir Butt-joint Weld. (3<sup>rd</sup> FSW Modelling and Material Flow Seminar, 2008-04-28, Geesthacht).

*Kostka, A.:* Microstructure and Properties of Al to Steel Friction Stir Overlap Welds. (7<sup>th</sup> International Friction Stir Welding Symposium, 2008-05-20 to 2008-05-22, Awaji Island, Japan).



- Kostka, A.:* Microstructure of Dissimilar Al to Steel Friction Stir Welds. (XIII International Conference on Electron Microscopy, 2008-06-08 to 2008-06-11, Zakopane, Poland).
- Kostka, A.:* Microstructure of dissimilar joints welded by FSW. (IPSUS Summer School 2008, 2008-09-08 to 2008-09-11, Hamburg-Bergedorf).
- Kostka, A.:* Electron microscopy in material characterization. (IPSUS Summer School 2008, 2008-09-08 to 2008-09-11, Hamburg-Bergedorf).
- Krein, R. and M. Palm:* The influence of Cr and B additions on the mechanical properties and oxidation behaviour of L21-ordered Fe-Al-Ti based aluminides at high temperature. (TMS Annual Meeting 2008, 2008-03-09 to 2008-03-13, New Orleans, USA).
- Kumar, P., M. E. Kassner, W. Blum and P. Eisenlohr:* New Observations on High Temperature Creep at Very Low Stresses. (Creep 2008, 2008-05-04 to 2008-05-09, Bad Berneck).
- Kumar, K. S., F. Stein and M. Palm:* Preliminary in-situ TEM observations of phase transformations in a Co-15 at.% Nb alloy. (Workshop: The Nature of Laves Phases XI, 2008-06-17, MPIE, Düsseldorf).
- Lange, B., C. Freysoldt and J. Neugebauer:* Band dispersion effects in supercell calculations for defects. (Computational Materials Science Workshop, 2008-08-25 to 2008-08-28, Schloss Eberburg, Bad Münster am Stein-Eberburg).
- Liu, T., D. Raabe and W. Mao:* Microtexture Evolution in Free-standing CVD Diamond Films: Growth and Twinning Mechanisms. (15<sup>th</sup> International Conference on the Texture of Materials (ICOTOM 15), 2008-06-01 to 2008-06-06, Carnegie Mellon University Center, Pittsburgh, USA).
- Liu, T., D. Raabe, S. Zaefferer and W. Mao:* On the Role of Nucleation during Microtexture Evolution in CVD Deposition of Diamond Thin Films. (15<sup>th</sup> International Conference on the Textures of Materials (ICOTOM 15), 2008-06-01 to 2008-06-06, Carnegie Mellon University Center, Pittsburgh, USA).
- Liu, T., D. Raabe, S. Zaefferer and W. Mao:* 3D EBSD Texture Study on CVD Diamond Films. (15<sup>th</sup> International Conference on the Texture of Materials (ICOTOM 15), 2008-06-01 to 2008-06-06, Carnegie Mellon University Center, Pittsburgh, USA).
- Lymperakis, L. and J. Neugebauer:* Thermodynamics and adatom kinetics of non-polar GaN surfaces. (Spring meeting of the German Physical Society (DPG), 2008-02-25 to 2008-02-29, Berlin).
- Lymperakis, L. and J. Neugebauer:* Ab-initio based calculation of GaN surfaces, interfaces and extended defects. (Colloquium Paul-Drude-Institut Berlin, 2008-05-22, Berlin).
- Lymperakis, L. and J. Neugebauer:* Thermodynamics and adatom kinetic on non-polar GaN surfaces: origin of a strong growth anisotropy. (E-MRS Spring meeting, 2008-05-26 to 2008-05-30, Strasbourg, France).
- Lymperakis, L. and J. Neugebauer:* Grain Boundaries: Statics and Mobility. (Computational Materials Science Workshop, 2008-08-25 to 2008-08-28, Schloss Eberburg, Bad Münster am Stein-Eberburg).
- Lymperakis, L. and J. Neugebauer:* Growth simulations of non-polar GaN surfaces: Thermodynamics, kinetics and dopant incorporations. (Bremen DFG Forschergruppe: Workshop in Riezlern, 2008-09-22 to 2008-09-27, Riezlern, Austria).
- Lymperakis, L. and J. Neugebauer (invited):* Ab initio study of Thermodynamics and adatom kinetics on non-polar GaN surfaces: Consequences on the growth morphology and the formation of nanowires. (International Conference on Nitride Semiconductors, 2008-10-06 to 2008-10-10, Montreux, Switzerland).
- Ma, D., M. Friák, J. Neugebauer and D. Raabe:* Novel multi-descriptive approach to crystal plasticity of textured beta-Ti alloys. (Spring meeting of the German Physical society (DPG), 2008-02-25 to 2008-02-29, Berlin).
- Ma, D., A. Counts, F. Roters, M. Friák, J. Neugebauer and D. Raabe:* Using ab initio Simulations in Texture Research. (15<sup>th</sup> International Conference on the Texture of Materials (ICOTOM 15), 2008-06-01 to 2008-06-06, Carnegie Mellon University Center, Pittsburgh, USA).
- Ma, D., D. Raabe, F. Roters, M. Friák and J. Neugebauer:* Modeling Rolling Textures of Beta Ti Alloys Using Constitutive Data From Ab-initio Simulations. (15<sup>th</sup> International Conference on the Texture of Materials (ICOTOM 15), 2008-06-01 to 2008-06-06, Carnegie Mellon University Center, Pittsburgh, USA).
- Ma, A., M. Friák, J. Neugebauer and D. Raabe (invited):* Ab initio based design of alloys. (MS&T'08, Symposium: Discovery and optimization of materials through computational design, 2008-10-05 to 2008-10-09, David Lawrence Convention Center, Pittsburgh, USA).
- Mardare, A. I., A. D. Wieck and A. W. Hassel:* High throughput synthesis and characterization of anodic oxides on valve metal combinatorial libraries. (2<sup>nd</sup> International IMPRS-SurMat Workshop on Surface and Interface Engineering in Advanced Materials, 2008-07-15 to 2008-07-16, Ruhr-Universität Bochum, Bochum).
- Mardare, A. I., A. Wieck and A. W. Hassel:* Combinatorial electrochemistry on valve metal alloys. (2<sup>nd</sup> International IMPRS-SurMat Workshop on Surface and Interface Engineering in Advanced Materials, 2008-07-15 to 2008-07-16, Ruhr-Universität Bochum, Bochum).



*Mardare, A. I., A. Ludwig, A. Savan, A. D. Wieck and A. W. Hassel:* Combinatorial microelectrochemistry with a scanning droplet cell on binary and ternary Ti, Ta and Hf alloys. (International Symposium on Anodizing Science and Technology 2008, 2008-07-23 to 2008-07-25, Rusutsu, Japan).

*Mardare, A. I., A. D. Wieck and A. W. Hassel:* Combinatorial microelectrochemistry using an automated scanning droplet cell. (59<sup>th</sup> Annual Meeting of the International Society of Electrochemistry, 2008-09-07 to 2008-09-12, Seville, Spanien).

*Mardare, A. I., A. D. Wieck and A. W. Hassel:* High Throughput Synthesis and Characterization of Ti Based Combinatorial Alloys. (7<sup>th</sup> International Symposium on Electrochemical Micro- and Nanosystems, 2008-09-07 to 2008-09-12, Ein-Gedi, Israel).

*Mardare, A. I., A. Ludwig, A. Savan, A. D. Wieck and A. W. Hassel:* High throughput growth and in situ characterization of anodic oxides on Ti, Ta and Hf combinatorial alloys. (Electrochemistry: Crossing Boundaries, GDCh, 2008-10-06 to 2008-10-08, Gießen).

*Marquardt, O., T. Hickel and J. Neugebauer:* Optical properties of semiconductor nanostructures, a PW-approach to real-space properties. (MRL seminar at UCSB, 2008-01-23, UCSB, Santa Barbara, USA).

*Marquardt, O., T. Hickel and J. Neugebauer:* Plane-wave implementation of the k.p-formalism including strain and piezoelectricity to study the optical properties of semiconductor nanostructures. (Spring meeting of the German Physical Society (DPG), 2008-02-25 to 2008-02-29, Berlin).

*Marquardt, O., T. Hickel and J. Neugebauer:* Optical properties of semiconductor nanostructures including strain and piezoelectric effects. (PARSEM meeting and workshop, 2008-03-26 to 2008-03-28, Cambridge, UK).

*Marquardt, O., T. Hickel and J. Neugebauer:* Neues Licht aus künstlichen Atomen - Halbleiterquantenpunkte. (Schulvortrag zur Jahrestagung der MPG, 2008-06-24 to 2008-06-27, Dresden).

*Marquardt, O., T. Hickel and J. Neugebauer:* Effect of strain and polarization on the electronic properties of 2-, 1- and 0-dimensional semiconductor nanostructures. (Computational Materials Science Workshop, 2008-08-25 to 2008-08-28, Schloss Eberburg, Bad Münster am Stein-Eberburg).

*Marquardt, O., T. Hickel and J. Neugebauer:* Modeling of electronic and optical properties of GaN/AlN quantum dots by using the k.p-method. (Bremen DFG Forschergruppe: Workshop in Riezlern, 2008-09-22 to 2008-09-26, Riezlern, Austria).

*Michalik, A. and M. Rohwerder:* Long range ion transport properties of conducting-polymers (59<sup>th</sup> Annual Meeting of the International Society of Electrochemistry, 2008-09-07 to 2008-09-12, Seville, Spain).

*Milenkovic, S., B. Bello Rodriguez and A. W. Hassel:* Novel self-organised Re nanostructures. (9<sup>th</sup> International Conference on Nanostructured Materials, 2008-06-01 to 2008-06-06, Rio de Janeiro, Brazil).

*Milenkovic, S., D. Frankel, A. J. Smith and A. W. Hassel:* Selective Phase Dissolution of NiAl-Mo Directionally Solidified Eutectic Alloys. (7<sup>th</sup> International Symposium on Electrochemical Micro- and Nanosystems, 2008-09-07 to 2008-09-12, Ein-Gedi, Israel).

*Moscicki, M., H. Pinto, A. Borbély and A. R. Pyzalla:* In-situ investigation of grain rotations during tensile straining of steel wires. (ICRS-8, 2008-08-06 to 2008-08-08, Denver, USA).

*Moscicki, M., H. Pinto, C. Paulmann, A. Borbély and A. R. Pyzalla:* In-Situ Investigation of Grain Rotations During Tensile Straining of Steel Wires. (MSE08 Materials Science and Engineering, 2008-09-01 to 2008-09-04, Nürnberg).

*Neugebauer, J. (invited):* Ab initio basiertes Computergestütztes Materialdesign: Von der chemischen Bindung zu realen Werkstoffeigenschaften. (Seminar at the TU Clausthal, 2008-01-24, Clausthal).

*Neugebauer, J. (invited):* Ab initio based modeling of engineering materials: From a predictive thermodynamic description to tailored mechanical properties. (UCSB Seminar, 2008-02-15, UCSB, Santa Barbara, USA).

*Neugebauer, J. (invited):* Ab initio design of engineering materials: Status and challenges. (UCSB-MPG Workshop on Inorganic Materials for Energy Conversion, Storage and Conservation, UCLA Lake Arrowhead Conference Center, 2008-02-19 to 2008-02-22, Lake Arrowhead, USA).

*Neugebauer, J. (invited):* Optimizing materials properties and epitaxial growth of semiconductor devices by ab initio based multiscale modeling. (Workshop: Molecular Modelling and Simulation in Applied Materials Science, 2008-03-10 to 2008-03-11, Frankfurt (Main)).

*Neugebauer, J. (invited):* Computational alloy design by ab initio based growth simulation. (9. Kinetikseminar der DGKK, 2008-03-12 to 2008-03-13, Aachen).

*Neugebauer, J. (invited):* Simulation of surface growth processes. (4<sup>th</sup> International Workshop on Crystal Growth Technology (IWCGT-4), 2008-05-18 to 2008-05-25, Beatenberg, Switzerland).

*Neugebauer, J. (invited):* Ab initio based modeling of engineering materials: From a predictive thermodynamic description to tailored mechanical properties. (Kolloquium der TUM, 2008-06-02, Garching).



*Neugebauer, J.* (invited): Materials design based on ab initio thermodynamics and kinetics. (Mid-term COST conference on Multiscale Modeling of Materials, 2008-06-25 to 2008-06-28, Brno, Czech Republic).

*Neugebauer, J.* (invited): Materials design based on ab initio thermodynamics and kinetics. (XVII International Materials Research Congress 2008, 2008-08-17 to 2008-08-21, Cancun, Mexico).

*Neugebauer, J.* (invited): Ab Initio Based Modeling of Engineering Materials: From a Predictive Thermodynamic Description to Tailored Mechanical Properties. (Materials Science and Engineering, 2008-09-01 to 2008-09-04, Nürnberg).

*Neugebauer, J.* (invited): Design of engineering materials based on ab initio thermodynamics and kinetics. (Materials Science and Technology 2008, 2008-10-05 to 2008-10-09, Carnegie Mellon University Center, Pittsburgh, USA).

*Neugebauer, J.* (invited): Ab initio based modeling of engineering materials: From a predictive thermodynamic description to tailored mechanical properties. (Multiscale Materials Modeling 2008, 2008-10-27 to 2008-10-31, Tallahassee, USA).

*Nikolov, S., D. Raabe, C. Sachs and H. Fabritius:* Hierarchical modeling of the mechanical properties of hard biological tissues: bone and lobster cuticle. (MU Conference, 2008-02-01, MPIE, Düsseldorf).

*Nikolov, S., C. Sachs, H. Fabritius, D. Raabe, M. Petrov, M. Friák, J. Neugebauer, L. Lymperekis and D. Ma:* Hierarchical modeling of the mechanical properties of lobster cuticle from nano- up to macroscale: The influence of the mineral content and the microstructure. (4<sup>th</sup> International Conference on Multiscale Materials Modeling, 2008-10-27 to 2008-10-31, Tallahassee, USA).

*Nikolov, S., M. Petrov, M. Friák, C. Sachs, H. Fabritius, D. Raabe, J. Neugebauer and L. Lymperekis* (invited): Multi-scale Modelling of the Elastic Properties of Biological Polymer-Based Nanocomposites with Hierarchical Microstructure: The Lobster Cuticle. (3<sup>rd</sup> International Conference on Polymer Behaviour, 2008-11-03 to 2008-11-06, Marrakesh, Morocco).

*Nnamchi, P., D. Ponge, D. Raabe, A. Barani, G. Bruckner and J. Krautshik:* Influence of the As-Cast Microstructure on the Evolution of the Hot Rolling Textures of Ferritic Stainless Steels with Different Compositions. (15<sup>th</sup> International Conference on the Textures of Materials (ICOTOM 15), 2008-06-01 to 2008-06-06, Carnegie Mellon University Center, Pittsburgh, USA).

*Özcan, Ö., A. T. Blumenau and G. Grundmeier:* Adsorption of Organosilanes on ZnO Surfaces. (2<sup>nd</sup> IMPRS-SurMat Workshop in Surface and Interface Engineering in Advanced Materials, 2008-07-14 to 2008-07-16, Ruhr-Universität Bochum, Bochum).

*Özcan, Ö., A. T. Blumenau and G. Grundmeier:* A combined experimental - computational approach: Revealing the organosilane to zinc oxide binding mechanism. (Euradh 2008 - Adhesion '08, 2008-09-03 to 2008-09-05, St Catherine's College, Oxford, UK).

*Özcan, Ö., G. Grundmeier and A. T. Blumenau:* Linking Stability Studies to the Preferential Growth of ZnO. (TACC2008, 2008-09-23 to 2008-09-27, Shanghai, China).

*Palm, M.* (invited): Fe-Al-based materials for structural applications at high temperatures. (ISPMA, 2008-08-24 to 2008-08-28, Prague, Czech Republic).

*Paninski, M., A. Drevermann, G. J. Schmitz, M. Palm, F. Stein, N. Engberding, M. Heilmaier, H. Saage, K. Kelm and S. Irsen:* Properties of cast Al-rich Ti-Al alloys. (TMS Annual Meeting 2008, 2008-03-09 to 2008-03-13, New Orleans, USA).

*Petrov, M., M. Friák, L. Lymperekis, D. Raabe and J. Neugebauer:* An ab-initio study of the hydrogen-bond network in crystalline alpha-chitin. (Spring meeting of the German Physical Society (DPG), 2008-02-25 to 2008-02-29, Berlin).

*Pfanner, G., C. Freysoldt and J. Neugebauer:* Numerical studies of the radial Schroedinger equation. (Computational Materials Science Workshop, 2008-08-25 to 2008-08-28, Schloss Ebernburg, Bad Münster am Stein-Ebernburg).

*Pinto, H., C. Barbatti, A. Guio, A. Jahn, J. Standfuß and A. R. Pyzalla:* Application of EBSD to the characterization of induction-assisted welds in high strength. (Electron Backscatter Diffraction Meeting, 2008-03-31 to 2008-04-01, University of Sheffield, Sheffield, UK).

*Pinto, H.* (invited): Diffraction studies of micro-structure and internal stresses in oxide scales. (DAAD Seminar New Materials, 2008-08-18 to 2008-08-20, UFRGS, Porto Alegre, Brazil).

*Pinto, H.* (invited): Welding metallurgy of steels. (VI-IPSUS Summer School 2008, 2008-09-08 to 2008-09-12, Hamburg-Bergedorf).

*Pinto, H.* (invited): Zerstörungsfreie Analyse der Eigenspannungen und der Schädigungen in Bauteilen mit Hilfe von Synchrotronstrahlung. (Industrieforum 2008: Neue Materialien im Licht der Zukunft, DESY, 2008-11-05, Hamburg).

*Ponge, D.:* Warmumformbarkeit von Stahl. (Kontaktstudium Werkstofftechnik Stahl, Teil III, Technologische Eigenschaften, Werkstoffausschuss im Stahlinstitut VDEh, TU Dortmund, 2008-06-22, Dortmund).

*Posner, R., K. Wapner, M. Stratmann and G. Grundmeier:* Hydrated Ion Transport at Polymer/Oxide/Metal-Interfaces in Non-Corrosive Atmosphere: Influence of Electric Field Gradients. (Gordon Conference Graduate Research Seminar on Aqueous Corrosion, Colby Sawyer College, 2008-07-19 to 2008-07-20, New London, USA).

*Posner, R. and G. Grundmeier:* Electrochemical and spectroscopic analysis of ion transport processes along polymer/oxide/metal interfaces in corrosive and non-corrosive atmosphere. (Euradh 2008 - Adhesion '08, St Catherine's College, 2008-09-03 to 2008-09-05, Oxford, UK).

*Prymak, O., F. Stein, G. Frommeyer and D. Raabe:* Rietveld refinement of site occupation in ternary Nb(Cr, Al)<sub>2</sub> Laves phase. (Workshop: The Nature of Laves Phases XI, 2008-06-17, MPIE, Düsseldorf).

*Prymak, O. and F. Stein:* Composition dependence of site occupancy and c/a ratio in hexagonal C14 Laves phase of the Nb-Cr-Al system. (TOFA Thermodynamics of Alloys 2008, 2008-06-22 to 2008-06-27, Krakow, Poland).

*Pyzalla, A. R. (invited):* In-situ Characterization of Damage Processes using Synchrotron Radiation. (International Conference: Advanced Processing for Novel Functional Materials, 2008-01-23 to 2008-01-25, Dresden).

*Pyzalla, A. (invited):* Werkstoffcharakterisierung durch Diffraktion und Tomographie mit Synchrotron-Röntgenstrahlung und Neutronen. (Symposium Hochleistungskeramik 2008, 2008-02-26 to 2008-02-27, TU Hamburg-Harburg, Hamburg).

*Pyzalla, A. (invited):* Sauropoden-Dinosaurier: Giganten aus nanokristallinem Material. (29. Adelbodener Werkstoffseminar des IWK 1 der Universität Karlsruhe, 2008-03-03 to 2008-03-06, Adelboden, Schweiz).

*Pyzalla, A. (invited):* Stahlforschung am MPIE - Tradition und Perspektiven. (Festkolloquium Prof. O. Pawelski, 75 Jahre, 2008-04-25, Stahlzentrum, Düsseldorf).

*Pyzalla, A. R., M. Dumont, N. Zoeger, C. Strelt, P. Wobrauscheck and M. Sander:* Synchrotron XRF analyses of element distribution in fossilized sauropod dinosaur bones. (Denver X-ray Conference, 2008-08-04 to 2008-08-08, Denver, USA).

*Pyzalla, A. R., A. Borbéy, A. Isaac, F. Sket, K. Dzieciol, M. di Michiel and T. Buslaps:* In-situ synchrotron tomography investigation of creep damage. (SPIE Optics + Photonics 2008, 2008-08-10 to 2008-08-14, San Diego, USA).

*Raabe, D., C. Sachs, H. Fabritius, P. Romano, L. Raue, H. Klein and A. Al-Sawalmih (invited):* Crystallographic Textures from the Exoskeleton of the Lobster Homarus Americanus and Calculation of the Mechanical Properties of the Calcite Phase. (15<sup>th</sup> International Conference on the Textures of Materials (ICOTOM 15), 2008-06-01 to 2008-06-06, Carnegie Mellon University Center, Pittsburgh, USA).

*Raabe, D., R. Degenhardt, R. Sellger, W. Klos, M. Sachtleber and L. Ernenputsch (invited):* Advances in the optimization of thin strip cast austenitic 304 stainless steel microstructures. (6<sup>th</sup> European Stainless Steel Conference, 2008-06-10 to 2008-06-13, Helsinki, Finland).

*Raabe, D., C. Sachs, H. Fabritius, M. Friák, M. Petrov and S. Nikolov (invited):* Principles to design and simulate hierarchical materials with property-relevant structural features. (Max-Planck-Institute for Colloids and Interfaces, 2008-07-07, Golm).

*Raabe, D. (invited):* Non equilibrium flow on gradient surfaces: Theory and experiment. (Sommer Workshop on Nano- & Microfluidics, 2008-07-30 to 2008-08-01, Bad-Honnef).

*Raabe, D.:* Large scale dynamics in a driven simple glass. (The XV<sup>th</sup> International Congress on Rheology, 2008-08-03 to 2008-08-08, Monterey, USA).

*Raabe, D., M. Friák, J. Neugebauer and A. Counts: (invited):* Homogenization in Polycrystal Mechanics on the Basis of First Principles Simulations. (IUTAM Symposium on Variational Concepts in Materials Mechanics, 2008-09-22, Ruhr-Universität Bochum, Bochum).

*Rablbauer, R. and G. Frommeyer:* Mechanical properties of ferritic stainless AlCr steels. (SCT 08 Steel in Cars and Trucks, 2008-06-01 to 2008-06-05, Wiesbaden).

*Rasp, W. and I. Jäckel:* Theoretische Untersuchung und industrielle Validierung des Querflusses beim Walzen auf flacher Bahn. (XXVII Verformungskundliches Kolloquium, 2008-01-01, Leoben, Austria).

*Raue, L. and D. Raabe:* Crystallographic and Topological Textures of Biological Materials and the Resulting Anisotropy of the Mechanical Properties. (15<sup>th</sup> International Conference on the Texture of Materials (ICOTOM 15), 2008-06-01 to 2008-06-06, Carnegie Mellon University Center, Pittsburgh, USA).

*Rohwerder, M.:* Role of Localized Protection for the Performance of Conducting Polymer Based Composite Coatings. (The 7<sup>th</sup> International Symposium on Electrochemical Micro- and Nanosystem Technologies (EMNT), 2008-09-15 to 2008-09-18, Ein-Gedi, Israel).

*Romano, P.:* Microstructure characterization of multiphase steels (TRIP) using EBSD techniques. (Corus Ceramics Research Centre, 2008-01-01, IJmuiden, The Netherlands).

*Romano, P., Y. Adachi and M. Wakita:* Morphology and thermal stability of ultra fine grained TRIP steels characterised by EBSD. (15<sup>th</sup> Electron Backscatter Diffraction Meeting, 2008-03-01, University of Sheffield, Sheffield, UK).



*Romano, P., S. Zaeferrer and D. Raabe:* Use of Orientation Gradients for the Identification of Different Microconstituents in Multiphase Steels. (15<sup>th</sup> International Conference on the Texture of Materials (ICOTOM 15), 2008-06-01 to 2008-06-06, Carnegie Mellon University Center, Pittsburgh, USA).

*Roters, F., L. Hantcherli and P. Eisenlohr:* Incorporating Twinning into the Crystal Plasticity Finite Element Method. (Materials Science and Engineering 2008, 2008-09-01 to 2008-09-04, Nürnberg).

*Sachs, C., S. Nikolov, H. H. Fabritius and D. Raabe:* Investigation and Modeling of the Elastic Properties of Lobster Cuticle Depending on its Grade of Mineralization. (MRS Spring Meeting, 2008-03-26, San Francisco, USA).

*Sachs, C., S. Yi and D. Raabe:* Investigation of the Lattice Strain Evolution in Tension and Compression of Different Phases in the Mineralized Lobster Cuticle. (MRS Spring Meeting, 2008-03-27, San Francisco, USA).

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*Santa, M., R. Posner and G. Grundmeier:* Surface enhanced Raman spectroscopy and Scanning Kelvin Probe studies of corrosive de-adhesion at polymer-metal interfaces. (The 59<sup>th</sup> Annual Meeting of the International Society of Electrochemistry, 2008-09-07 to 2008-09-12, Seville, Spain).

*Scharnberg, M., S. Kaps, V. Zaporozhchenko, S. Milenkovic, A. W. Hassel, F. Faupel and R. Adelung:* Mechanisms of the reflection of water jets on superhydrophobic surfaces. (Spring meeting of the German Physical Society (DPG), 2008-02-25 to 2008-02-29, Berlin).

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*Schmitt, M. and M. Spiegel:* Corrosion and fouling data of candidate materials for WtE components: Part II. (NextGen-BioWaste, 1<sup>st</sup> Progress Meeting 2008, 2008-02-01, Schiphol Airport, Amsterdam, The Netherlands).

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*Senöz, C., A. Majus, M. Rohwerder and W. Schuhmann:* Microstructural and Surface Potential Study of Al-4 wt% Cu-Mg (DURAL) Alloy. (ICAA 11, 11<sup>th</sup> International Conference on Aluminium Alloys, 2008-09-22, Aachen).

*Silva, P. A.:* Hot Direct Extrusion of Abrasion Resistant Fe-base Metal Matrix Composites - Interface Characterization and Mechanical Properties of Co-Extruded Layered Structures. (DGM International Symposium: Friction, Wear and Wear Protection, 2008-04-09 to 2008-04-11, Aachen).

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*Sket, F., K. Dzieciol, A. Isaac, G. Sauthoff, A. Borbély and A. R. Pyzalla:* In-Situ Investigation of Creep and Creep Damage Using Synchrotron Microtomography. (Workshop on X-Ray Micro Imaging of Materials, Devices and Organisms, 2008-10-22 to 2008-10-24, Dresden).

*Smith, A. J. and A. W. Hassel:* Direct Manipulation of Nano Wires with Nanotweezers for mechanical testing, electrical and spectroscopic characterization. (Bunsentagung 2008, 2008-05-01 to 2008-05-03, Saarbrücken).

*Song, J.:* Explosive Cladding of Titanium onto Low Carbon Steel. (International SurMat Workshop, 2008-07-15 to 2008-07-16, Department of Material Science and Engineering, Ruhr-Universität Bochum, Bochum).

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*Stein, F.:* Composition dependence of nanohardness and Young's modulus in diffusion couples containing Laves phases. (Workshop: The Nature of Laves Phases X, 2008-01-10, Dresden).

*Stein, F. and O. Prymak:* The variation of the c/a ratio in hexagonal Laves phases. (Workshop: The Nature of Laves Phases X, 2008-01-10, Dresden).

*Stein, F., G. Frommeyer and S. Schneider:* Processing of eutectic NiAl-Cr and NiAl-Re alloys under microgravity. (Meeting: TEMPUS Parabolic Flight September 2007, 2008-01-16, Bonn).

*Stein, F. and G. Frommeyer:* High-performance soft Magnetic iron-silicon alloys for industrial applications processed under microgravity. (Space for Innovation - Industry Forum for Material Research and Microgravity; Fachtagung: Materialforschung und Schwerelosigkeit für Industrieanwendungen, 2008-05-08, MPIE, Düsseldorf).

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*Stein, F., O. Prymak, O. Dovbenko and M. Palm:* Phase equilibria of Laves phases in ternary Nb-X-Al systems with X = Cr, Fe, Co. (Discussion Meeting on Thermodynamics of Alloys - TOFA 2008, 2008-06-22 to 2008-06-27, Krakow, Poland).

*Stein, F. and S. Voss:* The nature of Laves phases: Observation of defect softening. (Materials Science and Engineering MSE 2008, 2008-09-01 to 2008-09-04, Nürnberg).

*Stein, F., G. Frommeyer and S. Schneider:* Eutectic NiAl-Re alloys processed under microgravity. (Meeting: TEMPUS Parabolic Flight April 2008, 2008-11-10, Bonn).

*Stratmann, M. (invited):* Tailored oxides for controlling ionic mobility at buried materials. (UCLA Workshop, 2008-02-19 to 2008-02-22, Lake Arrowhead, USA).

*Stratmann, M., A. W. Hassel and M. Rohwerder (invited):* Microelectrochemical Investigations of Interfaces and Surfaces of Advanced Materials. (7<sup>th</sup> International Symposium on Electrochemical Micro- and Nanosystems, 2008-09-07 to 2008-09-12, Ein-Gedi, Israel).

*Stratmann, M. (invited):* In-situ SKP Study of the Diffusion of Ions along Buried Interfaces. (214<sup>th</sup> Meeting of ECS — The Electrochemical Society - Corrosion Division Uhlig Award Talk, 2008-10-12 to 2008-10-17, Honolulu, USA).

*Struss, J., N. Znidarsic, A. Ziegler and S. Hild:* Microscopic anatomy and mineral composition of cuticle in amphibious isopods *Ligia italica* and *Titanethes albus* (Crustacea: Isopoda). (European Microscopy Congress EMC 2008, 2008-09-01 to 2008-09-15, Aachen).

*Sturm, D., M. Heilmaier, H. Saage, M. Paninski, G. J. Schmitz, M. Palm, F. Stein, A. Drevermann, N. Engberding, K. Kelm and S. Irsen:* Creep strength of centrifugally cast Al-rich TiAl alloys. (11<sup>th</sup> International Conference on Creep and Fracture of Engineering Materials and Structures, 2008-05-04 to 2008-05-09, Bayreuth).

*Swaminathan, S., M. Spiegel and M. Rohwerder:* Effect of annealing conditions on the selective oxidation of quaternary model alloy. (4<sup>th</sup> International Conference on Diffusion in Solids and Liquids, 2008-07-09, Barcelona, Spain).

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*Thissen, P., B. Özkaya, J. Wieland and G. Grundmeier:* Analytical Studies of the Formation of Silver Nanoparticles in Layer by Layer Deposited Polyelectrolyte Films. (Euradh 2008 - Adhesion '08, 2008-09-03 to 2008-09-05, St Catherine's College, Oxford, UK).

*Todorova, M. and J. Neugebauer:* A new approach to obtain electrochemical E/pH diagrams derived from the viewpoint of semiconductor defects. (Spring meeting of the German Physical Society (DPG), 2008-02-25 to 2008-02-29, Berlin).

*Todorova, M. and J. Neugebauer:* Towards an ab initio description of corrosion. (International Workshop on Ab initio Description of Iron and Steel (ADIS2008), 2008-06-15 to 2008-06-20, Schloss Ringberg, Kreuth).

*Todorova, M. and J. Neugebauer:* Towards an ab initio description of corrosion. (3<sup>rd</sup> Computational Materials Science Workshop, 2008-08-25 to 2008-08-28, Schloss Ebernburg, Bad Münster am Stein-Ebernburg).



*Torres, E., P. U. Biedermann and A. T. Blumenau:* A DFT study of Alkanethiol adsorption sites on Au(111) surfaces. (2<sup>nd</sup> IMPRS-SurMat Workshop in Surface and Interface Engineering in Advanced Materials, 2008-07-14 to 2008-07-16, Ruhr-Universität Bochum, Bochum).

*Torres, E., P. U. Biedermann and A. T. Blumenau:* The Role of Gold Adatoms in Self-Assembled Monolayers of Thiol on Au(111). (6<sup>th</sup> Congress of the International Society for Theoretical Chemical Physics, ISTCP-VI, 2008-07-19 to 2008-07-24, University of British Columbia, Vancouver, Canada).

*Torres, E., P. U. Biedermann and A. T. Blumenau:* High density structures of ethyl-thiol SAM's on Au(111): A DFT study. (SurMat Seminar, 2008-08-12 to 2008-08-14, Schloß Gnadenenthal, Kleve).

*Udyansky, A., M. Friák and J. Neugebauer:* An ab-initio study of the phase transitions in the interstitial Fe-C solid solutions. (Spring meeting of the German Physical Society (DPG), 2008-02-25 to 2008-02-29, Berlin).

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*Uijtewaal, M., T. Hickel and J. Neugebauer:* Phase transformations of Ni<sub>2</sub>MnGa shape memory alloy from first principles. (Spring meeting of the German Physical Society (DPG), 2008-02-25 to 2008-02-29, Berlin).

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*Uijtewaal, M., T. Hickel and J. Neugebauer (invited):* Understanding the phase transformations of the Ni<sub>2</sub>MnGa shape memory system. (2008-10-09, Nijmegen, The Netherlands).

*Valtiner, M. and G. Grundmeier (invited):* Towards a deeper understanding of molecular adhesion mechanisms by a combined approach of single molecule adhesion and DFT studies. (23. Workshop: Novel Materials and Superconductors, 2008-02-23 to 2008-03-01, Universitätssportheim Plannersalm, Donnersbach, Austria).

*Valtiner, M. and G. Grundmeier:* Hydroxide adsorption on ZnO(0001) surfaces. (15<sup>th</sup> Vienna2k workshop, 2008-03-26 to 2008-03-29, Vienna, Austria).

*Valtiner, M. and G. Grundmeier (invited):* Acidic dissolution mechanism, pH-dependent stabilization and adhesion of single molecules on single crystalline ZnO(0001)-Zn model surfaces studied by in-situ AFM and DFT simulation. (PSI-k Summerschool for Modern Concepts for Creating and Analyzing Surfaces and Nanoscale Materials, 2008-05-12 to 2008-05-16, Sant Feliu de Guixols, Spain).

*Valtiner, M. and G. Grundmeier:* Acidic dissolution mechanism, pH-dependent stability and adhesion of single molecules studied on single crystalline ZnO(0001)-Zn model surfaces by in-situ AFM studies. (Gordon Conference Graduate Research Seminar on Aqueous Corrosion, 2008-07-19 to 2008-07-20, Colby Sawyer College, New London, USA).

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*Van Caenegem, N., K. Verbeken, R. Petrov, N. M. van der Pers and Y. Houbaert:* Shape Recovery and  $\epsilon$  -  $\gamma$  Transformation in  $\text{Fe}_{29}\text{Mn}_7\text{Si}_5\text{Cr}$  SMA. (3<sup>rd</sup> International Conference: Smart Materials, Structures and Systems, 2008-06-13 to 2008-06-16, Acreale, Italy).

*Varnik, F. and D. Raabe:* Stability and kinetics of droplets: A free energy based lattice Boltzmann study. (Spring Meeting German Physical Society (DPG), 2008-02-25 to 2008-02-29, Berlin).

*Varnik, F.:* Flows driven by wettability gradients: A lattice Boltzmann study. (Spring meeting of the German Physical Society (DPG), 2008-02-25 to 2008-02-29, Berlin).

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*Varnik, F. and D. Raabe:* Stability and kinetics of droplets. (The 5<sup>th</sup> International Conference for Mesoscopic Methods in Engineering, 2008-06-16 to 2008-06-20, Amsterdam, The Netherlands).

*Varnik, F. and D. Raabe:* Lattice Boltzmann studies of confined flows at intermediate Reynolds numbers: The role of wall roughness. (The 5<sup>th</sup> International Conference for Mesoscopic Methods in Engineering, 2008-06-16 to 2008-06-20, Amsterdam, The Netherlands).

*Varnik, F.:* Lattice Boltzmann simulations of multiphase and multicomponent systems. (Institut für Experimentalphysik, Universität Augsburg, 2008-07-02, Augsburg).

*Varnik, F.:* Some micro- and nanofluidic issues using a free energy based lattice Boltzmann approach: Finite size driven droplet evaporation and wetting dynamics on chemical gradients. (Max-Planck-Institut für Metallforschung, 2008-07-03, Stuttgart).

*Varnik, F. and D. Raabe:* Finite size driven droplet evaporation and kinetics of droplets: A lattice Boltzmann study. (Summer Workshop on Nano- & Microfluidics, 2008-07-30 to 2008-08-01, Bad-Honnef).

*Varnik, F. and D. Raabe:* Stability and kinetic of droplets: A free energy based lattice Boltzmann study. (Sommer Workshop on Nano- & Microfluidics, 2008-07-30 to 2008-08-01, Bad-Honnef).

*Varnik, F.:* Diffusion and structural relaxation in sheared glassy systems. (The XV<sup>th</sup> International Congress on Rheology, 2008-08-03 to 2008-08-08, Monterey, USA).

*Venzlaff, H., D. Enning, F. Widdel, M. Stratmann and A. W. Hassel:* Microbial corrosion induced by a highly aggressive SRB strain. (2<sup>nd</sup> International IMPRS-SurMat Workshop on Surface and Interface Engineering in Advanced Materials, 2008-07-15 to 2008-07-16, Ruhr-Universität Bochum, Bochum).

*Venzlaff, H., F. Widdel, M. Stratmann and A. W. Hassel:* Microbial corrosion induced by a new highly aggressive SRB strain. (59<sup>th</sup> Annual Meeting of the International Society of Electrochemistry, 2008-09-07 to 2008-09-12, Seville, Spanien).

*Verbeken, K., L. Barbé and D. Raabe:* Crystallographic characterization of a phosphorus added TRIP steel. (15<sup>th</sup> international conference on textures of materials (ICOTOM15), 2008-06-01 to 2008-06-06, Carnegie Mellon University Center, Pittsburgh, USA).

*Verbeken, K., L. Bracke, L. Kestens and J. Penning:* Microstructural and Textural Evolutions in a Cold Rolled High Mn TWIP Steel. (15<sup>th</sup> International Conference on the Texture of Materials (ICOTOM 15), 2008-06-01 to 2008-06-06, Carnegie Mellon University Center, Pittsburgh, USA).

*Vogel, S. C., M. Eumann, M. Palm and F. Stein:* Investigation of the crystallographic structure of the  $\epsilon$  phase in the Fe-Al system by high-temperature neutron diffraction. (American Conference on Neutron Scattering (ACNS 2008), 2008-05-11 to 2008-05-15, Santa Fe, USA).

*Vogel, S. C., M. Eumann, M. Palm and F. Stein:* Investigation of the crystallographic structure of the  $\epsilon$  phase in the Fe-Al system by high-temperature neutron diffraction. (20<sup>th</sup> Annual Rio Grande Symposium on Advanced Materials 2008, 2008-09-24, Albuquerque, USA).

*von Pezold, J. and J. Neugebauer:* Molecular Dynamics simulation of dislocations in metals. (German-Korean miniworkshop, 2008-01-31, MPIE, Düsseldorf).

*von Pezold, J. and J. Neugebauer:* Hydrogen enhanced local plasticity - An atomistic study. (Spring meeting of the German Physical Society (DPG), 2008-02-25 to 2008-02-29, Berlin).

*von Pezold, J. and J. Neugebauer:* Multiscale simulations of Hydrogen embrittlement (MSE 08, 2008-09-01 to 2008-09-04, Nürnberg).

*Voss, S., F. Stein and D. Raabe:* The mechanical properties of Nb-TM Laves phases - sample preparation and the composition dependence of the microhardness in Nb-Fe Laves phases. (Workshop: The Nature of Laves Phases XI, 2008-06-17, MPIE, Düsseldorf).

*Wang, X. and G. Grundmeier:* Thin multifunctional silver/fluorocarbon plasma polymer nanocomposite films on metals. (The 9<sup>th</sup> International Conference on Nanostructured Materials, 2008-06-01 to 2008-06-06, Rio de Janeiro, Brazil).



*Weber, F., I. Schestakow, D. Raabe and F. Roters:* Investigation of texture and microstructure in a bent monocrystalline Cu-nanowire using EBSD and crystal plasticity finite element simulations. (7<sup>th</sup> GAMM Seminar on Microstructures, 2008-01-25 to 2008-01-26, Bochum).

*Weber, S., B. Özkan and W. Theisen:* Protective Gas Sintering of Wear Resistant Composite Materials. (MSE08 Materials Science and Engineering, 2008-09-01 to 2008-09-04, Nürnberg).

*Weber, S. and P. Silva:* Diffusion in Fe-Base Thick Coatings Produced by Hot Extrusion. (Thermo-Calc Anwendertreffen, 2008-09-11 to 2008-09-12, Aachen).

*Wielant, J., R. Posner, R. Hausbrand, G. Grundmeier and H. Terry:* Surface and bulk properties of oxide films and their influence on the adhesion and delamination of organic coatings. (213<sup>th</sup> ECS Meeting, 2008-05-18 to 2008-05-25, Phoenix, USA).

*Winning, M.:* 3D EBSD measurements in ultra fine grained Cu 0.17wt% Zr obtained from ECAP. (Carnegie Mellon University Center, 2008-02-26, Pittsburgh, USA)

*Winning, M. and D. Raabe:* Influence of Grain Boundary Mobility on Texture Evolution during Recrystallization. (15<sup>th</sup> International Conference on the Texture of Materials (ICOTOM 15), 2008-06-01 to 2008-06-06, Carnegie Mellon University Center, Pittsburgh, USA).

*Woldemedhin, M. T., D. Raabe and A. W. Hassel:* Evaluation of Surface reactivity of  $\beta$ -Ti. (2<sup>nd</sup> International IMPRS-SurMat Workshop on Surface and Interface Engineering in Advanced Materials, 2008-07-15 to 2008-07-16, Ruhr-Universität Bochum, Bochum).

*Wu, G. and S. Zaefferer:* Development of a TEM-based Orientation Microscopy System and its Application to Study of Microstructure and Texture of Nanocrystalline NiCo Samples. (15<sup>th</sup> International Conference on the Texture of Materials (ICOTOM 15), 2008-06-01 to 2008-06-06, Carnegie Mellon University Center, Pittsburgh, USA).

*Zaafarani, N., F. Roters, S. Zaefferer and D. Raabe:* Tomographic 3D EBSD Analysis of Deformation and Rotation Patterns around Nanoindentations. (15<sup>th</sup> International Conference on the Texture of Materials (ICOTOM 15), 2008-06-01 to 2008-06-06, Carnegie Mellon University Center, Pittsburgh, USA).

*Zaefferer, S. (invited):* Application of 3-dimensional orientation microscopy to study the microstructure of different heavily deformed metals. (Plasticity 2008, 2008-01-08, Kona, USA).

*Zaefferer, S. (invited):* New insights into deformed microstructures by 3D-EBSD based orientation microscopy. (TMS 2008, 2008-03-11, New Orleans, USA).

*Zaefferer, S., S. Ishikawa and F. Stein:* Distinction of different Laves phase types by EBSD in a TiCr diffusion couple: Robust detection of subtle differences in EBSD patterns. (Electron Backscatter Diffraction Meeting 2008, RMS Conference, 2008-03-31 to 2008-04-02, University of Sheffield, Sheffield, UK).

*Zaefferer, S. (invited):* SEM and TEM-based orientation microscopy and Monte-Carlo modelling: A toolbox to study recrystallisation nucleation processes. (Electron Backscatter Diffraction Meeting 2008, RMS Conference, 2008-03-31 to 2008-04-02, University of Sheffield, Sheffield, UK)

*Zaefferer, S.:* SEM and TEM-based orientation microscopy and Monte-Carlo modelling: A toolbox to study recrystallisation nucleation processes (Institut für Magnesium-Forschung, Forschungszentrum Geesthacht, 2008-04-07, Geesthacht)

*Zaefferer, S. (invited):* SEM and TEM-based orientation microscopy and Monte-Carlo modelling: A toolbox to study recrystallisation nucleation processes. (Annual Meeting of the Canadian Microscopical Society 2008, 2008-05-22, Montreal, Canada).

*Zaefferer, S.:* Investigations into deformation mechanisms and damage of TRIP and TWIP steels, (University of British Columbia, Institute for Materials Science, 2008-05-26, Vancouver, Canada)

*Zaefferer, S.:* Application of 3-dimensional orientation microscopy to study the microstructure of different heavily deformed metals. (University of British Columbia, Institute for Materials Science, 2008-05-27, Vancouver, Canada)

*Zaefferer, S.:* Orientation microscopy in the TEM – techniques, possibilities and limits (Lecture at the Institute for Materials Science, Lehigh University, 2008-05-29, Bethlehem, USA).

*Zaefferer, S.:* Study on the Recrystallisation Nucleation Mechanisms of Heavily Deformed Fe36% Ni by High Resolution 3-Dimensional Orientation Microscopy. (15<sup>th</sup> International Conference on the Texture of Materials (ICOTOM 15), 2008-06-01 to 2008-06-06, Carnegie Mellon University Center, Pittsburgh, USA).

*Zaefferer, S., S. Ishikawa and F. Stein (invited):* Distinction of Different Laves Phase Types by EBSD in a TiCr Diffusion Couple: Robust Detection of Subtle Differences in EBSD Patterns. (Workshop: The Nature of Laves Phases XI, 2008-06-17, MPIE, Düsseldorf).

*Zaefferer, S.:* Application of 3-dimensional orientation microscopy to study the microstructure of different heavily deformed metals (Centre of Electron Nanoscopy, Danish Technical University Copenhagen, 2008-07-10, Copenhagen, Denmark)



*Zambaldi, C., S. Zaefferer and S. Wright:* Determination of texture and microstructure of ordering domains in gamma-TiAl. (Electron Backscatter Diffraction Meeting by the Royal Microscopical Society, 2008-03-31 to 2008-04-01, University of Sheffield, Sheffield, UK).

*Zambaldi, C., S. Zaefferer and F. Roters:* Order domains in intermetallic TiAl - EBSD characterization and crystal plasticity modeling. (GLADD meeting, University of Gent, 2008-04-25, Gent, Belgium).

*Zambaldi, C., S. Zaefferer, F. Roters and D. Raabe:* Micro-mechanical implications of TiAl order domains. (The annual plenary meeting of the EU sixth framework programme IMPRESS integrated project, 2008-05-26, Camogli, Italy).

*Zambaldi, C., S. I. Wright and S. Zaefferer:* Determination of Texture and Microstructure of Ordering Domains in gamma-TiAl. (15<sup>th</sup> International Conference on the Texture of Materials (ICOTOM 15), 2008-06-01 to 2008-06-06, Carnegie Mellon University Center, Pittsburgh, USA).

*Zeller, S., J. Gnauk and G. Frommeyer:* The production of rapidly solidified thin wires using in-rotating-liquid-spinning. (Advanced Processing for Novel Functional Materials - APNFM 2008, 2008-01-23 to 2008-01-25, Dresden).

*Ziegler, A. and S. Hild:* Distribution and function of amorphous CaCO<sub>3</sub> and Calcite within the tergite cuticle of terrestrial isopods (Crustacea). (European Microscopy Congress EMC 2008, 2008-09-01 to 2008-09-15, Aachen).

*Zuo, J., P. Keil, M. Valtiner, P. Thissen and G. Grundmeier:* Ag nanostructures on fluoroalkylsilane self assembled monolayers: The dependence on perfluoroalkyl chain length. (The 9<sup>th</sup> International Conference on Nanostructured Materials, 2008-06-01 to 2008-06-06, Rio de Janeiro, Brazil).



# Publications

2007

## Books, Book Chapters and Editorial Work

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*Varnik, F.:* Diffusion, structural relaxation and rheological properties of a simple glass forming model: A molecular dynamics study. 5th International Workshop on Complex Systems, Sendai, Japan, 2007. (Eds.: Tokuyama, M., Oppenheim, I., Nishiyama, H.) Proceedings/ AIP Conference Mathematical and Statistical Physics, Huntington, USA (2008).

*Varnik, F.:* Can microscale wall roughness trigger unsteady/chaotic flows? 5th International Workshop on Complex Systems, Sendai, Japan, 2007. (Eds.: Tokuyama, M., Oppenheim, I., Nishiyama, H.) Proceedings/ AIP Conference Mathematical and Statistical Physics, Huntington, USA (2008).

*Verbeken, K., Barbé, L. and Raabe, D.:* Crystallographic characterization of a phosphorus added TRIP steel. (15<sup>th</sup> international conference on textures of materials (ICOTOM15), Carnegie Mellon University Center, Pittsburgh, USA) (2008).

*Verbeken, K., Bracke, L., Kestens, L. and Penning, J.:* Microstructural and Textural Evolutions in a Cold Rolled High Mn TWIP Steel. (15<sup>th</sup> International Conference on the Texture of Materials (ICOTOM 15), Carnegie Mellon University Center, Pittsburgh, USA) (2008).



# Habilitation, Doctoral, Diploma, Master and Bachelor Theses

## Habilitation Theses

### 2007

*PD Dr. Winning, M.:* Grain Boundary Mechanics: Interactions between mechanical stresses and grain boundaries – New approaches to microstructure control and materials design. RWTH Aachen, Aachen (2007).

*PD Dr. Varnik, F.:* Some selected issues on computer simulations of complex systems: Structural relaxation, external forces and hydrodynamics. RWTH Aachen, Aachen (2007).

## Doctoral Theses

### 2007

*Dr.-Ing. Al-Sawalmih, A.:* Crystallographic Texture of the Arthropod Cuticle Using Synchrotron Wide Angle X-ray Diffraction. RWTH Aachen, Aachen (2007).

*Dr.-Ing. Barani, A. A.:* Optimization of the Critical Content of Tramp Elements in Ultra-High Strength Silicon Chromium Spring Steels through Thermomechanical Treatment. RWTH Aachen, Aachen (2007).

*Dr.-Ing. Bastos da Silva Fanta, A.:* Characterization of the Microstructure, Grain Boundaries and Texture of Nanostructured Electrodeposited CoNi by use of EBSD. RWTH Aachen, Aachen (2007).

*Dr.-Ing. Brück, U.:* Mikrostrukturen und Eigenschaften ultrahochfester Leichtbaustähle auf der Basis Fe-Mn-Al-C. TU Clausthal, Clausthal-Zellerfeld (2007).

*Dr.-Ing. Detroy, S.:* Charakterisierung und Entwicklung von bainitischen Multiphasen-Stählen. RWTH Aachen, Aachen (2007).

*Dr.-Ing. Frommert, M.:* Dynamische Rekristallisation unter konstanten und transienten Umformbedingungen. RWTH Aachen, Aachen (2007).

*Dr.-Ing. Godara, A.:* Polymer crystallization and micromechanical behavior of fiber-reinforced polymer composites – a microstructural view on advanced polymer composites. RWTH Aachen, Aachen (2007).

*Dr.-Ing. Goerdeler, M.:* Application of a dislocation density based flow stress model in the integrative through-process modeling of Aluminium production. RWTH Aachen, Aachen (2007).

*Ph.D. McLaughlin, K.:* Ph.D. defense at Cambridge University, Cambridge, UK (2007).

*Dr.-Ing. Siska, F.:* Continuum vs. discrete dislocation dynamics modeling of mechanical behaviour of thin metallic films. University of Groningen, Groningen (2007).

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*Dr. rer. nat. Swaminathan, S.:* Selective surface oxidation and segregation upon short term annealing of model alloys and industrial steel grades. Ruhr-Universität Bochum, Bochum (2007).

*Dr.-Ing. Walczak (formerly Stempniewicz), M.:* Release Studies on Mesoporous Microcapsules for New Corrosion Protection Systems. Ruhr-Universität Bochum, Bochum (2007).

*Dr.-Ing. Wang, X.:* Thin Functional Plasma Polymer and Metal/Plasma Polymer Nanocomposite Films. Ruhr-Universität Bochum, Bochum (2007).

*Dr.-Ing. Yusupov, A.:* Berechnung der Breitung und der Breitenform beim Warmflachwalzen von Stahl. TUBA Freiberg, Freiberg (2007).



## 2008

- Dr.-Ing. Agudo, L.:* Influence of alloying on the microstructure and mechanical properties of steel-aluminum-joints produced by metal arc joining with special focus on the intermetallic phase seam. RWTH Aachen, Aachen (2008).
- Dr.-Ing. Coelho, R.:* Joining of light-weight materials by friction stir welding and laser beam welding. Ruhr-Universität Bochum, Bochum (2008).
- Dr. Cedat, D.:* Etudes expérimentale et numérique du comportement mécanique d'un composite métal – céramique: MoTo C 30%, Ecole Centrale Paris, Paris, France (2008).
- Dr. rer. nat. Dick, A.:* Ab initio STM and STS simulations on magnetic and nonmagnetic metallic surfaces. University of Paderborn, Paderborn (2008).
- Dr.-Ing. Klüber, C.:* Korrelation von mechanischen Eigenschaften und Kristallorientierung auf mikroskopischer und nanoskopischer Ebene. RWTH Aachen, Aachen (2008).
- Dr. rer. nat. Lill, K. A.:* Electrochemical Investigations on the Corrosion Properties of New Classes of Light Weight Steels. Ruhr-Universität Bochum, Bochum (2008).
- Dr.-Ing. Neelakantan, L.:* Surface Engineering of Nickel Titanium Shape Memory Alloys. Ruhr-Universität Bochum, Bochum (2008).
- Dr.-Ing. Raue, L.:* Kristallographische Texturen und richtungsabhängige mechanische Eigenschaften des Exoskeletts des amerikanischen Hummers sowie Texturen weiterer Biomaterialien. RWTH Aachen, Aachen, 2008).
- Dr.-Ing. Sachs, C.:* Microstructure and mechanical properties of the exoskeleton of the lobster *Homarus americanus* as an example of a biological composite material. RWTH Aachen, Aachen (2008).
- Dr. rer. nat. Smith, A. J.:* Electrochemical Processing of Directionally Solidified Eutectic Alloys: Release and Characterization of Metallic Nanowires., Heinrich-Heine-Universität, Düsseldorf (2008).
- Dr.-Ing. Thomas, I.:* Untersuchung metallphysikalischer und messtechnischer Grundlagen zur Rekristallisation und Erholung mikrolegierter IF Stähle., RWTH Aachen, Aachen, 2008).
- Ph.D. Tjahjanto, D.:* Micromechanical Modeling and Simulations of Transformation-Induced Plasticity in Multiphase Carbon Steels. University Delft, Delft, The Netherlands, 2008).
- Dr.-Ing. Zaaferani, N.:* Experimental and theoretical investigation of nanoindentation in a Cu single crystal. RWTH Aachen, Aachen (2008).

## Diploma Theses

### 2007

- Dönecke, K.:* Untersuchungen zum Korrosionsverhalten von FeAlCr-Leichtbaustählen. Heinrich-Heine-Universität, Düsseldorf (2007).
- Engberding, N.:* Der Einfluss des Legierungselements Phosphor auf die mechanischen Eigenschaften mikrolegierter TWIP-Stähle. FH Osnabrück, Osnabrück (2007).

### 2008

- Aydin, U.:* Ab initio Studie der Energetik und Dynamik von Wasserstoff in Metallen. Technische Fachhochschule Berlin, Berlin (2008).
- Gomoll, T.:* Ab initio Berechnung von Phononenspektren in Systemen mit reduzierter Symmetrie. Technische Fachhochschule Berlin, Berlin (2008).
- Uchdorf, T.:* Developing a general purpose database application for multiphysics. Fachhochschule Aachen, Standort Jülich (2008).



## Master Theses

### 2007

*Kim, O.:* Ab-initio study of formation and interaction energies in steel and their relations to the solubility limit of carbon in austenite and ferrite. RWTH Aachen, Aachen (2007).

*Ma, D.:* Phase Transformations and Elastic Properties of Ti-Nb and Ti-Mo Binary Alloys: Experiments in Conjunction with Ab-initio Calculations and Finite Element Simulations RWTH Aachen, Aachen (2007).

*Voß, S.:* Untersuchungen zur Mikrostruktur und den temperaturabhängigen mechanischen Eigenschaften von warmverformten intermetallischen Fe<sub>3</sub>Al (Cr,Ni)-Basislegierungen mit D0<sub>3</sub> Struktur. Technische Universität Clausthal, Clausthal-Zellerfeld (2007).

### 2008

*Bubnik, V.:* Visualization and Modeling of Molecules and Crystals. Brno University of Technology, Brno, Czech Republic (2008).

*Yuan, L.:* High resolution EBSD investigation of deformation induced martensitic transformation in TRIP steel and Austenitic stainless steel. RWTH Aachen, Aachen (2008).

## Bachelor Theses

### 2007

*Weber, F.:* Metallphysikalische Untersuchungen der Plastizität von Einkristallen in mikroskopischen Dimensionen unter Verwendung von FEM Simulationen. FH Düsseldorf, Düsseldorf (2007).

*Sander, B.:* Gefüge- und Eigenschaftsuntersuchungen an den Ti-Mo<sub>20</sub>-Zr<sub>7</sub>-Ta<sub>5</sub> und Ti-Nb<sub>35</sub>-Zr<sub>7</sub>-Ta<sub>5</sub>  $\beta$  Titanlegierungen für medizinische Implantate. FH Düsseldorf, Düsseldorf (2007).

*Schestakow, I.:* Experimentelle Untersuchung kristallographischer Aspekte der Rekristallisation von reinem Magnesium. FH Düsseldorf, Düsseldorf (2007).

### 2008

*Bobrowski, U.:* Wärmebehandlung und Charakterisierung rascherstarrer Fe<sub>71</sub>Si<sub>10</sub>B<sub>15</sub>Nb<sub>3</sub>Cu<sub>1</sub>-Legierungen. Universität Duisburg-Essen, Duisburg (2008).

*Buchwalter, F.:* Herstellung und Charakterisierung Al-legierter gezogener perlitischer Drähte von extremer Festigkeit. Fachhochschule Bonn-Rhein-Sieg, Rheinbach (2008).

*Budak, M.:* Einfluss der Al<sub>3</sub>Ti-Phase auf die Phasensaumdicke bei Diffusionsversuchen mit festem Titan und flüssigem Aluminium. Universität Duisburg-Essen, Duisburg (2008).

*Hölzer, S.:* Physikalische und mechanische Eigenschaften hoch Mn- und Al-legierter austenitischer TRIPLEX-Stähle. FH Düsseldorf, Düsseldorf (2008).

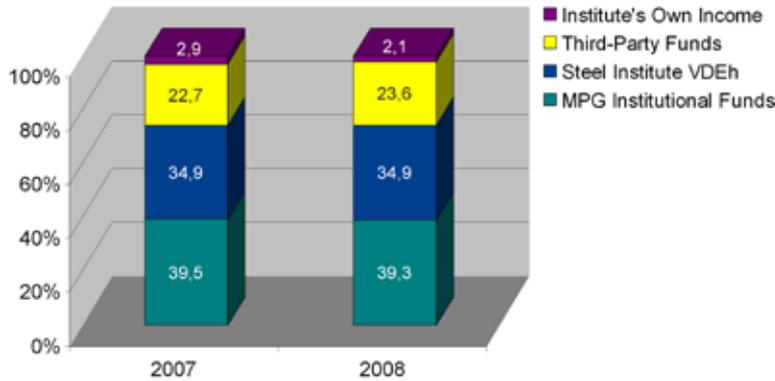
*Marinkovic, R.:* Herstellung von Si Nanostrukturen durch gerichete Erstarrung und elektrochemische Prozessierung, FH Bonn-Rhein-Sieg, Rheinbach (2008).



# Budget of the Institute

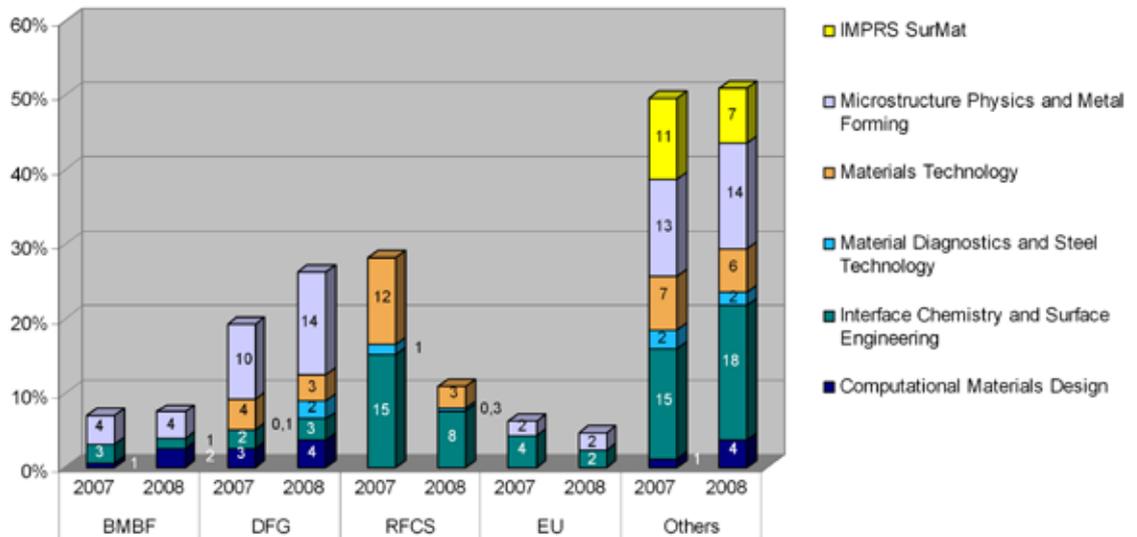
## Revenue

(percentual contributions to total revenue without appointment-related investment funds and general reconstruction of the buildings; year 2008 data estimated)



## Third-Party Funds

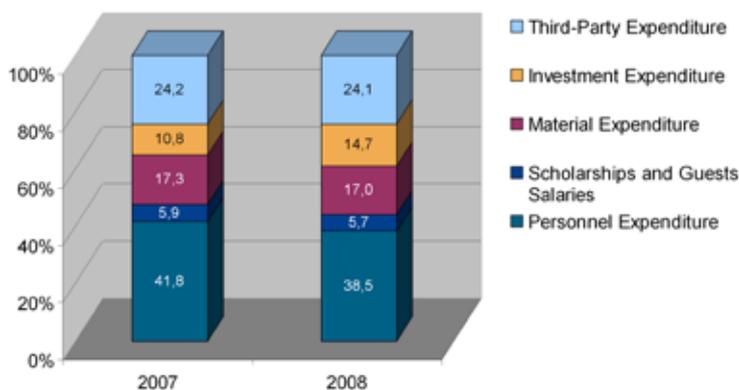
(percentual contributions to total revenue including personnel, material, investments; year 2008 data estimated)



BMBF: Ministry of Science and Education  
 DFG: German Science Foundation  
 RFCS: Research Fund for Coal and Steel  
 EU: European Union

## Expenditure

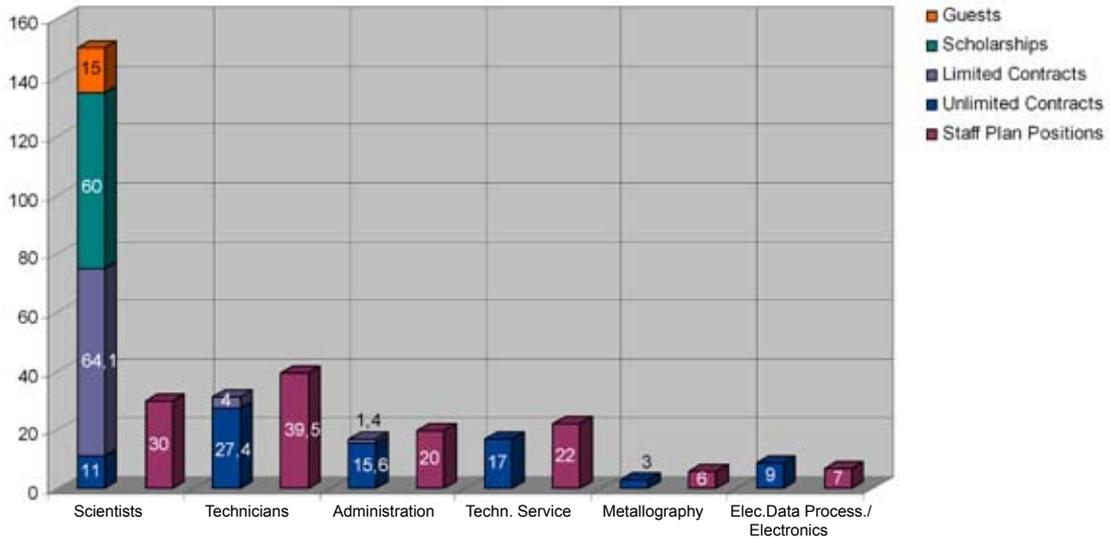
(percentual distribution of total expenditure; investments include large-scale apparatus, electronic data processing, appointment-related investments, separate investment for basic equipment; year 2008 data estimated)



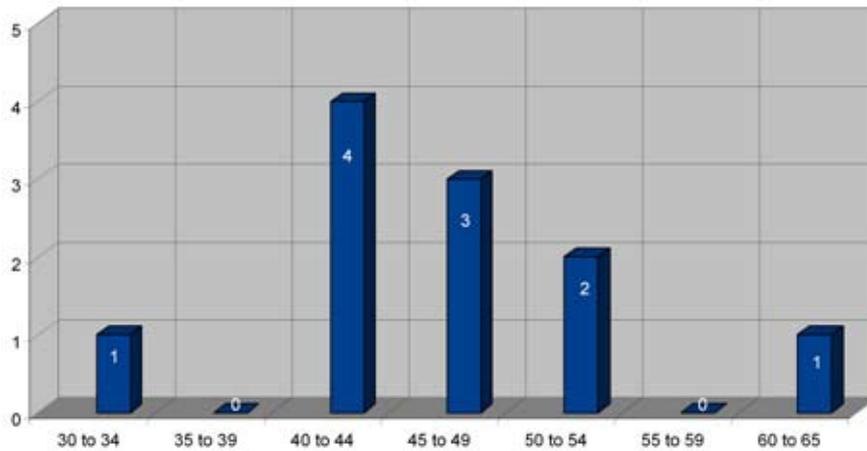


# Personnel Structure

**Number of Occupied Scientific / Non-Scientific Positions compared with Staff Plan Positions** (Dec. 2008, estimated)

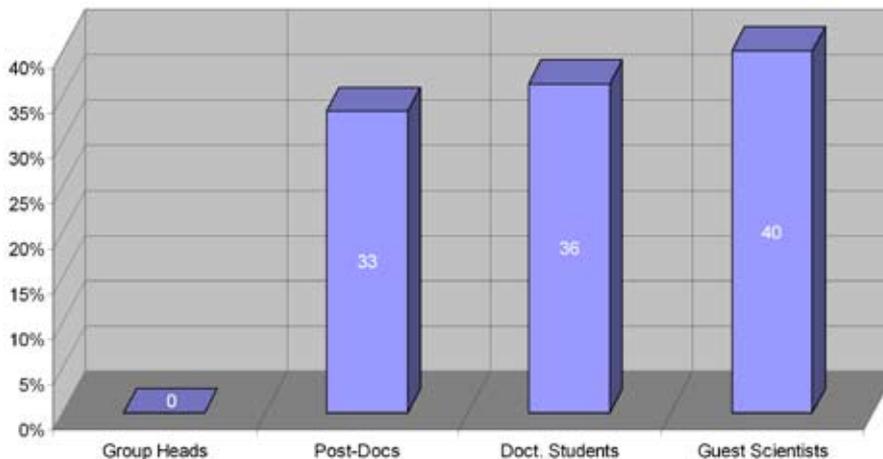


**Age Distribution: Number of Scientists with Unlimited Contracts** (Dec. 2008, estimated)



## Female Scientists

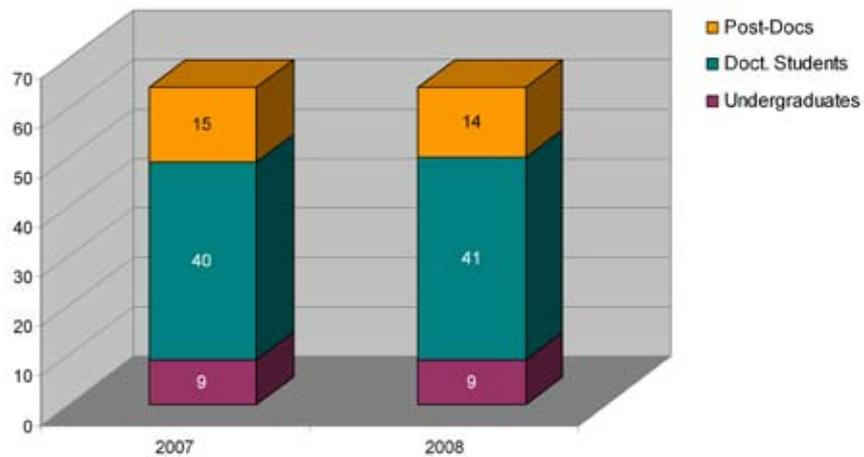
(Dec. 2008, estimated, percentual numbers)





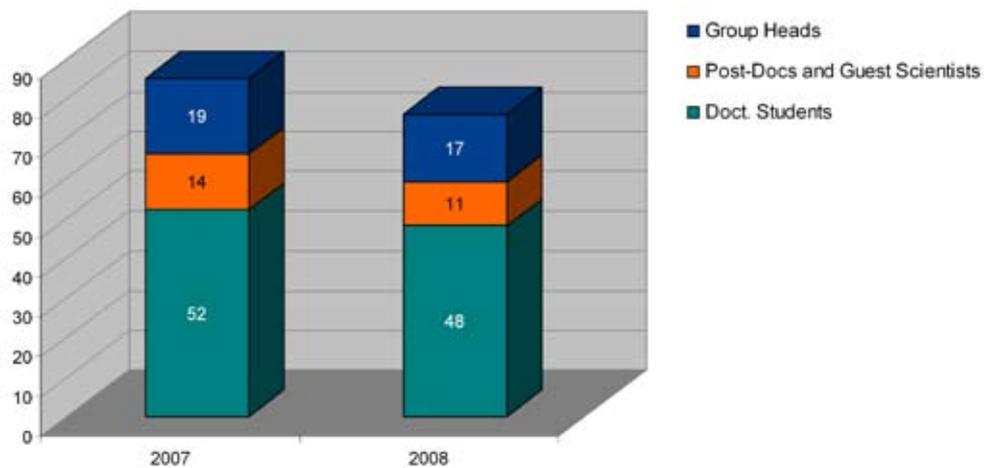
### Number of Junior Scientists Financed via Third-Party Funds

(year 2008 data estimated)



### Number of Junior Scientists not Financed via Third-Party Funds

(year 2008 data estimated)





# The Institute in Public

Date	Description
2007	Frontcover and Calendar Picture of the week, MPG Calendar (A. W. Hassel)
2008-04-26- 2008-10-05	Exhibition in the ‚Deutsches Technikmuseum Berlin‘ MAX PLANCK Revolutionär wider Willen: Contribution of the CM-Department of the MPIE (V. Hasse and T. Hickel)
2008-05-17- 2008-05-25	Ideenpark Stuttgart, Contribution on the Functional principle of the shape memory effect as part of the presentation of the ICAMS institute (T. Hickel and J. Neugebauer)
2008-08	Frontcover of Advanced Engineering Materials Vol. 10, No. 8 (F. Weber)

Repeatedly newspapers and online journals reported on the work in the Max-Planck-Institut für Eisenforschung:

## 2007

Date	Journal	Title
2007-01-02	www.automobil-industrie.de	Neue Stahllegierungen steigern Motorleistung
2007-03-25	Welt am Sonntag online	Ein Institut zur Eisenforschung
2007-03-25	Welt am Sonntag online	Eiserne Lady mit Humor
2007-05-23	DFG Pressemitteilungen	DFG bewilligt elf neue Sonderforschungsbereiche
2007-05-24	Idw-online.de	RWTH Aachen und das Max-Planck-Institut entwickeln eine neue Klasse von Strukturwerkstoffen
2007-05-23	BerliNews	Neue Sonderforschungsbereiche 2007
2007-06-11	Idw-online.de	Neue DFG-Forschergruppe an der Ruhr-Universität: Mikrostrukturen analysieren und modellieren
2007-06-11	Ruhr-Universität Bochum: Presseinformationen	Bochumer Ingenieure weiter im Aufwind: Neue DFG-Forschergruppe an der Ruhr-Universität ‚Analysis and computation of microstructure in finite plasticity‘
2007-06-28	Kölner Stadtanzeiger	Die zwölf Max-Planck-Institute in NRW
2007-07	Technology Review	Stahl aus dem Computer
2007-07	Arcelor Mittal Report	Knochenarbeit für die Stahlforschung
2007-08	Stahl und Eisen	Netzwerk Stahltechnologie als Chance für die Zukunft
2007-09	International Careers Report, AAAS-Science Business Office Feature	A Strategy for the Future
2007-09-04	www.mpg.de	Intelligenter Stahl für das Auto von morgen
2007-09-04	www.uni-protokolle.de	Super-Stahl für sichere Autos



2007-09-05	Rheinische Post	Forscher entwickeln neuen Stahl für die Zukunft
2007-09-05	www.wilantis.de	Super-Stahl für supersichere Autos
2007-09-05	auto-presse.de	Neuer Karosserie-Stahl macht Autos sicherer
2007-09-05	www.all4engineers.com	Superplastischer Stahl für sichere Autos
2007-09-06	www.djnewsletters.de	Forscher entwickeln Stahl für sichere Autos
2007-09-07	www.autoservicepraxis.de	Max-Planck-Institut forscht an Stahl-Karosserien
2007-09-10	www.young-germany.de	New super steel for safer cars
2007-09-11	Dowjones VDW News	Forscher entwickeln Stahl für sichere Autos
2007-09-12	Weltonline	Elastischer Super-Stahl für sichere Autos
2007-09-12	www.recyclingmagazin.de	Forscher entwickeln Stahl für sichere Autos
2007-10	Technology Review	Hart wie Gummi
2007-10	www.blechnet.com	Stahlboom und Billig-Konkurrenz dominieren Jahreskonferenz des international Iron and Steel Institute
2007-10	Das Industriemagazin	Stahlboom und Billig-Konkurrenz bestimmen Weltstahl-Kongress
2007-10	Stahl und Eisen	Superstahl für sichere Autos
2007-10	Stahl und Eisen	Wissenschaftler berechnen mit Hilfe der Quantenmechanik den Stahl von morgen
2007-10	www.bbr.de	Take it IISI
2007-10-05	VDI Nachrichten	„Super-Stahl“ bringt noch mehr Sicherheit ins Auto
2007-10-10	Financial Times	Schwere Schlitten leicht gemacht
2007-10-28	Welt am Sonntag	Keine Idee darf verloren gehen
2007-11	Stahl	Quantenmechanik für den Stahl von morgen
2007-11	Stahl	Super-Stahl für sichere Autos
2007-11	Stahlreport	Konzept Kommunikation
2007-11	Geschmiedeter Stahl, Stahl-Zentrum	Die Zukunft nimmt schon heute Formen an
2007-11	stahl und eisen	Steel in motion
2007-11-07	Rheinische Post	Branche in Goldgräberstimmung
2007-11-07	Tagesspiegel	Härter als Stahl
2007-11-07	Weltonline	Das Auto aus dem Modulbaukasten
2007-11-07	Rheinische Post Stahl	Intelligenter Aufprallschutz
2007-11-22	idw-online.de	Bauteile maßgeschneidert- Clausthaler Sonderforschungsbereich 675 im 6. Industriekolloquium vorgestellt
2007-12	Bänder Bleche Rohre	Take it IISI



## 2008

Date	Journal	Title
2008-01	Polskie Towarzystwo Chemiz- ne	Prof. Dr. Hans-Jürgen Engell (1925-2007), an Obituary
2008-01	blechnet	Intelligenter Stahl von morgen
2008-01-14	Industrieanzeiger	Stahl verstehen lernen
2008-01-21	MM Maschinenmarkt Indus- triemagazin	Umformeigenschaften frühzeitig beeinflussen
2008-02-18	Industrieanzeiger	Stahl zehrt sich für die Sicherheit auf
2008-02-18	MM Maschinenmarkt Indus- triemagazin	Intelligenter Stahl für Autos von morgen
2008-02-18	Industrieanzeiger	Werkstoff-Quantensprung aus dem Supercomputer
2008-03	Brigitte woman	Ich wollte forschen, das ist alles
2008-03-17	Weltonline	Bedeutender Sprung in der Werkstoffentwicklung
2008-03-17	Weltonline	Spitzenposition im Nano-Mikro-Bereich
2008-03-18	www.derstandard.de	Hart und immer härter
2008-04-25	VDI Nachrichten	Leichtere Autos dank legierter Stähle
2008-05	Transfermagazin	Dicker Fisch: ICAMS-Institut für Materialforschung
2008-05	Transfermagazin	Max-Planck-Institut für Eisenforschung – Grundlegend und praxisnah
2008-06	steel research int.	Oskar Pawelski 75 Years
2008-06	FTD	Moleküleschieben am Computer
2008-06-04	Arte Experiment Wissen	Max-Planck Sonderausstellung im Deutschen Technik- museum Berlin
2008-06-05	Westdeutsche Zeitung	Magnesiumspäne gehen in Flammen auf
2008-06-05	Rheinische Post	Magnesiumspäne: Arbeiter verhindert größeren Brand
2008-06-06	Ingenieur-Werkstoffe	Supraduktile und hochfeste Leichtbaustähle für den Zukünftigen Fahrzeugbau
2008-06-06	idw-online.de	Materialforschungszentrum ICAMS an der Ruhr-Uni- versität offiziell eröffnet
2008-06-06	www.pm.ruhr-uni-bochum.de	ICAMS an der Ruhr-Universität offiziell eröffnet
2008-06-06	ldw-online.de	Europaweit einzigartiges Materialforschungszentrum – ICAMS an der Ruhr-Universität Bochum nimmt Arbeit auf
2008-06-06	www.thyssenkrupp-steel.com	ICAMS an der Ruhr-Universität offiziell eröffnet
2008-06-08	ruhrFAN	Materialforschungszentrum ICAMS nimmt seine Arbeit auf
2008-06-17	www.ftd.de	Moleküleschieben am Computer



2008-06-17	idw-online.de	Zuwahl neuer Mitglieder: 23 Wissenschaftler verstärken Acatech
2008-06-27	VDI Nachrichten	Innovative Materialentwicklung findet am Computer statt
2008-07	stahl und eisen	Werkstoffentwicklung aus dem Computer
2008-07-21	www.inar.de	Konferenz: Automobilbau – Trends in der Füge- und Werkstofftechnik
2008-07-22	Die Welt	Neue Stahlsorten vom Computer-Reißbrett
2008-08-25	Handelblatt	Stahl aus dem Rechner
2008-09	Stahl	Neuen Herausforderungen begegnen
2008-09-15	Stahl und Eisen	Leicht, fest und extrem dehnbar
2008-10-14	Ruhr-uni-Bochum Presseinformation	ICAMS legt in der Lehre los: Dreifache Antrittsvorlesung in der RUB, Werkstoffsimulation zwischen den Disziplinen
2008-10-19	Finanztreff.de	FTD: Autoblech – hart und weich zugleich
2008-11-13	Welt	Stahl Report: Der Hummer als Vorbild

## Film

Date	Broadcasting Station	Title
2007-09-20	3SAT	Nano: Stahlerfinder
2007-09-29	ORF	Newton: Smart Driving – Intelligenter Stahl für die Autos von morgen
2008-10-16	WDR	Planet Wissen: Stahl- altes Eisen mit glänzender Zukunft