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PART I.

THE INSTITUTE

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Management of the Institute

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

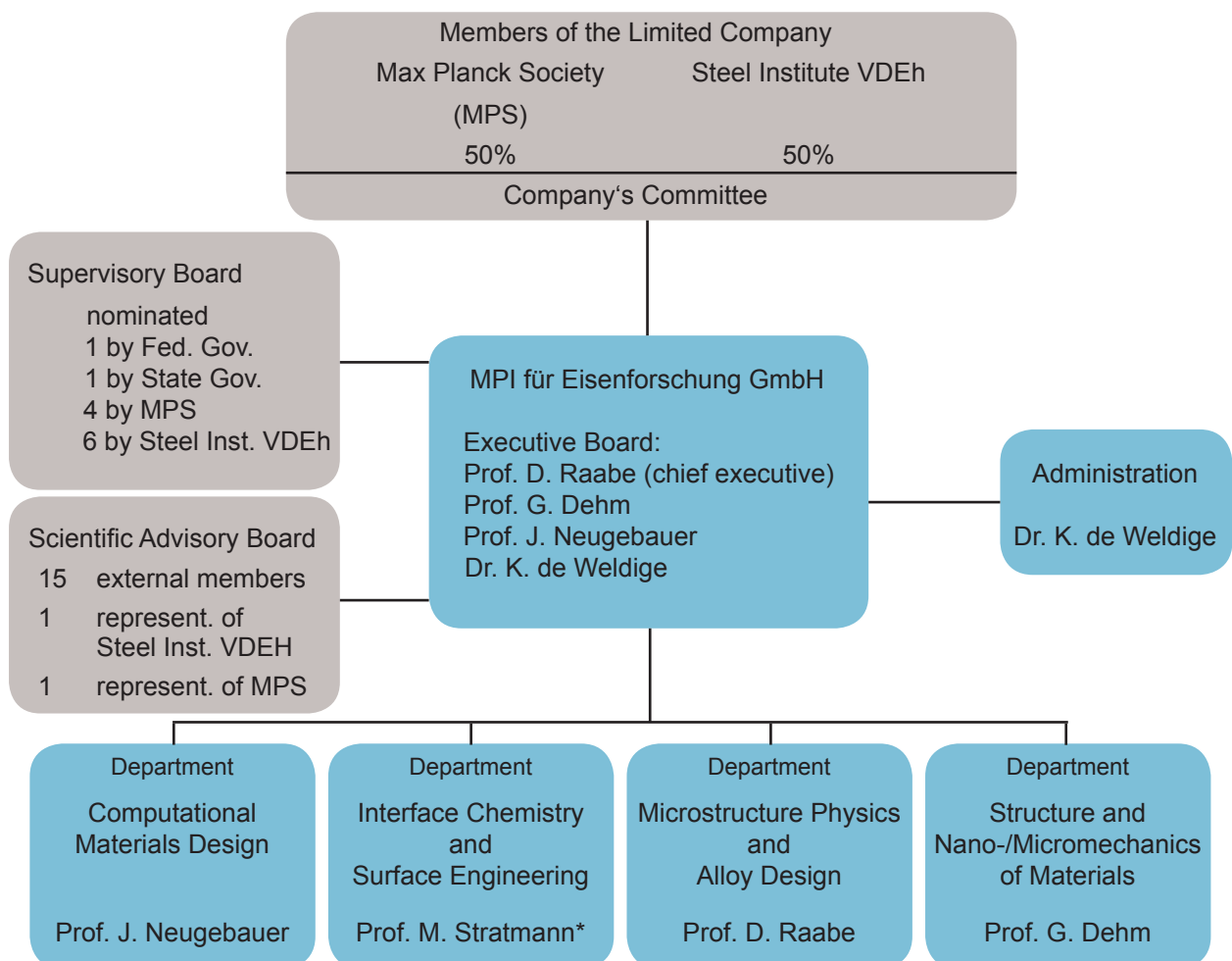
The Institute was founded in 1917 by the Verein Deutscher Eisenhüttenleute (VDEh) and incorporated into the Kaiser-Wilhelm-Gesellschaft, the predecessor of the Max Planck Society. The MPIE was first located in Aachen and was associated with the Technical University of Aachen. Later, in 1934/35, the Institute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

In 1946, the Institute's heavily damaged buildings were reconstructed, work resumed and the MPIE was integrated into the newly formed Max Planck Society in 1948. 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. Since then it has operated on the legal basis of a limited liability company (GmbH) and its budget is covered by the Steel Institute VDEh and the Max Planck Society.

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 and the administrative director of the Institute form an executive board of directors. The position of a managing director is filled, in rotation, by one of the board members. A board, which supervises the MPIE's activities, consists of representatives from the federal government, the state of North Rhine-Westphalia, 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.

MAX-PLANCK-INSTITUT FÜR EISENFORSCHUNG GMBH
Management and Organization (Nov. 2015)



* Prof. M. Stratmann is on leave. Temporary head of the department is Prof. J. Neugebauer.



Scientific Organization

The MPIE conducts basic research on metallic alloys and related materials. It pursues an approach where material systems are studied under consideration of their highly complex underlying nanostructures on the one hand and their exposure to extreme environmental conditions on the other hand.

The Institute is divided into four 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 (on leave); temporary: Prof. J. Neugebauer): aspects of environmentally accelerated degradation of surfaces and interfaces such as corrosion and deadhesion and the engineering of new and stable surfaces and interfaces
- Microstructure Physics and Alloy Design (Prof. D. Raabe): alloy design and mathematical modelling of microstructures and properties during processing and their experimental investigation using microscopy, atom probe tomography, and diffraction methods
- Structure and Nano-/Micromechanics of Materials (Prof. G. Dehm): mechanical response of materials focusing on small length scales by applying nano-/micromechanical approaches combined with advanced microstructure characterization techniques

Each department is subdivided into research groups which are typically managed by group heads. The figure on page 12 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.

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 all departments.

In concerted activities, scientific and technological breakthroughs in highly competitive research areas are achieved. Selected scientific highlights including such interdepartmental research activities are described in Part III which is divided into the five topics

- Development of new structural materials
- Analysis of microstructure-related materials properties
- Analysis and enhancement of the stability of surfaces and interfaces
- Development of scale-bridging simulation of materials
- Enhancement of energy materials

For each of these five 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 three years.

In summary, the research within the MPIE is organized vertically in highly specialized departments and research groups and horizontally in interdepartmental 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, a homogeneous research profile and an intense interdepartmental cooperation.

Service groups provide the scientific departments with valuable expertise. These services include the production, processing and testing 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, a computer network centre and a research coordination office.



Scientific Scopes of the Departments

Department Computational Materials Design Prof. J. Neugebauer	Department Interface Chemistry and Surface Engineering Prof. M. Stratmann*	Department Microstructure Physics and Alloy Design Prof. D. Raabe	Department Structure and Nano-/Micromechanics of Materials Prof. G. Dehm
Ab Initio Calculation of Thermodynamic and Kinetic Data	Electrochemical Surface Science	Alloy Design and Synthesis	Micro-/Nanomechanics and -tribology of Materials and Interfaces
Theoretical Studies on Microstructure Properties and Dynamics	Catalysis and Corrosion	Micromechanics	In situ Methods Interlinking Mechanics and Microstructure
Computer Aided Alloy Design	Adhesion and Functional Coatings, Surfaces and Interfaces	Correlative Atom Probe Tomography	Advanced Transmission Electron Microscopy
Multiscale Modelling	Fundamental Research on Surface and Coating Related Process Technology	Segregation Engineering and Interface Manipulation	Thin Films, Nanostructured Materials and Intermetallics

* Prof. M. Stratmann is on leave. Temporary head of the department is Prof. J. Neugebauer.

Interdepartmental Research Activities

Department Computational Materials Design Prof. J. Neugebauer	Department Interface Chemistry and Surface Engineering Prof. M. Stratmann*	Department Microstructure Physics and Alloy Design Prof. D. Raabe	Department Structure and Nano-/Micromechanics of Materials Prof. G. Dehm
Development of new structural materials			
Analysis of microstructure-related materials properties			
Analysis and enhancement of the stability of surfaces and interfaces			
Development of scale-bridging simulation of materials			
Enhancement of energy materials			

* Prof. M. Stratmann is on leave. Temporary head of the department is Prof. J. Neugebauer.



Scientific Groups and Departments (2015)

Computational Materials Design Prof. J. Neugebauer	Interface Chemistry and Surface Engineering Prof. M. Stratmann*	Microstructure Physics and Alloy Design Prof. D. Raabe	Structure and Nano-/Micromechanics of Materials Prof. G. Dehm	Independent Max Planck Research Groups
Ab Initio Thermodynamics Dr. M. Friak (until Sep. 2013)	Atomistic Modelling Dr. S. Wippermann (since Feb. 2013)	Adaptive Structural Materials Dr. C. Tasan	Advanced Microstructure Characterization Dr. C. Liebscher (since Apr. 2015)	Nanoanalytics and Interfaces Prof. C. Scheu (since Apr. 2014)
Adaptive Structural Materials Dr. B. Grabowski	Christian Doppler Lab. for Diffusion and Segregation Mechanisms Dr. M. Rohwerder (until Dec. 2014)	Alloy Design and Thermomechanical Processing Dr. D. Ponge	Intermetallic Materials Dr. M. Palm Dr. F. Stein	External Members and Fellows
Computational Phase Studies Dr. T. Hickel	Corrosion Dr. M. Rohwerder	Alloys for Additive Manufacturing Dr. E. Jägler (since Apr. 2015)	Nano-/ Micro-mechanics of Materials Dr. C. Kirchlechner (since Mar. 2013)	External Scientific Members Prof. M. Hillert Prof. R. Kirchheim
Computer Centre Dr. C. Freysoldt	Electrocatalysis Dr. K. J. J. Mayrhofer	Atom Probe Tomography Dr. P. Choi	Nanotribology Dr. S. Brinckmann (since Dec. 2014)	Max Planck Fellow High Temperature Materials Prof. G. Eggeler (until Dec. 2014)
Defect Chemistry and Spectroscopy Dr. C. Freysoldt	Interaction Forces and Functional Materials Dr. M. Valtiner	Biological Composites Dr. H. Fabritius	Synthesis of Nanostructured Materials N.N.	Max Planck Fellow Self-Reporting Materials Prof. J. Schneider (since Oct. 2015)
Electrochemistry and Corrosion Dr. M. Todorova (since Oct. 2015)	Interface Spectroscopy Dr. A. Erbe	Combinatorial Metallurgy and Processing Dr. H. Springer	Max Planck Research Group High Temperature Materials Prof. G. Eggeler (since Jan. 2015)	
Mesoscale Simulation Dr. R. Spatschek (until Oct. 2015)	Interface Structures and High-Temperature Materials Dr. F. Renner (until Jun. 2013)	Interface Design of Solar Cells Dr. O. Cojocaru-Mirédin (until Sep. 2015)		
ERC Starting Grant TIME BRIDGE Dr. B. Grabowski (since Jul. 2015)		Microscopy and Diffraction Dr. S. Zaefferer		
		Theory and Simulation Dr. F. Roters		
		Coop. with RWTH Aachen University Interface Design in Solar Cells Dr. O. Cojocaru-Mirédin (since Sep. 2015)		
Scientific Service Groups				
Processing Dr. H. Springer	Metallography Dr. S. Zaefferer	Materials Testing Dr. D. Ponge	Research Coordination Office Dr. K. Hübel	

* Prof. M. Stratmann is on leave. Temporary head of the department is Prof. J. Neugebauer.



Recent Developments

From 2012 to 2015 several major structural, scientific and administrative developments were made to further strengthen the Institute's scientific profile

The recently appointed director and executive G. Dehm started his department focusing on "Structure and Nano-/ Micromechanics of Materials". The department was opened in October 2012 and the major construction efforts, devoted particularly to hosting many of the new advanced electron microscopy facilities, will be essentially finished by mid 2016. The department works on nano- and micromechanics – mainly performed *in situ* using electron microscopy and x-ray diffraction, advanced electron microscopy characterization, nanotribology, synthesis of nano-structured and high-temperature intermetallic materials.

Also a new independent research group on "Nanoanalytics and Interfaces" has been opened by C. Scheu who joined the MPIE in April 2014. The main research activities include the atomic and electronic structure investigations of defects and interfaces in materials used for renewable energy applications. The group has initiated first collaborations with the groups of *in-situ* nano-/micromechanics and advanced electron microscopy in the department of G. Dehm, atom probe tomography and alloy design in the department of D. Raabe, electrochemical studies in the department of M. Stratmann, and plans to correlate experimentally determined bonding characteristics (via EELS) with those calculated in the department of J. Neugebauer.

The new team members complement the activities of the existing departments on "Microstructure Physics and Alloy Design" (D. Raabe), "Interface Chemistry and Surface Engineering" (M. Stratmann (on leave), temporary heads: M. Rohwerder & J. Neugebauer), and "Computational Materials Design" (J. Neugebauer). Together, the four departments cover a broad range of expertise and tools for designing, synthesizing, and analyzing complex structural materials and associated processes under harsh and reactive environmental conditions. More specific, strong intramural collaborations with the new department, exploiting particularly its electron optical and *in-situ* experimental expertise, have been established in the fields of deformation-driven phase transformations, development of ductile and tough metallic glasses, hydrogen embrittlement, strengthening mechanisms in metallic nanolaminate composites, nanostructured

thermoelectric materials, chemical decomposition in martensitic alloys, density reduced high strength steels, correlative atom probe and transmission electron microscopy and complexions to name but a few of the latest projects jointly pursued. A series of inter-departmental workshops has been established to foster collaborations including also young team members and to identify new promising joint research topics of high scientific risk, gain and synergy.

In the period reviewed (end of 2012 - 2015) several new research groups have been initiated, namely, the group on "Nanotribology" by S. Brinckmann, the BMBF-funded group on "Interface Design in Solar Cells " by O. Cojocar-Mirédin, a newly awarded ERC Starting Grant group by B. Grabowski on time-scale bridging potentials for realistic molecular dynamics simulations, a group funded by the joint Max-Planck-Fraunhofer initiative on "Alloys for Additive Manufacturing" by E. Jäggle, the group for "Nano-/Micromechanics of Materials" by C. Kirchlechner, the group for "Advanced Microstructure Characterization" by C. Liebscher, and the "Atomistic Modelling" group as well as the BMBF NanoMatFutur research group „Semiconducting nanocomposites with tailored electronic and optical properties for solar energy conversion“ by S. Wippermann.

A number of large-scaled and networking projects were initiated or prolonged during the last years. Examples are the European research network on „Mechanics of Nano-Objects“ (MECANO) which is concerned with the mechanical properties of materials in small dimensions or the collaboration with the Interdisciplinary Centre for Advanced Materials Simulation (ICAMS) at the Ruhr-Universität Bochum. ICAMS focuses on the development and application of a new generation of simulation tools for multi-scale materials modelling.

Other initiatives include the ERC Advanced Grant funded joint group on "Adaptive Structural Materials" which jointly develops novel experimental and theoretical tools and methodologies, employing them to understand micro-mechanisms governing macro-properties and design property-optimized novel structural alloys.

The Institute pursues also several large joint initiatives with its neighbour universities RWTH Aachen University (SFB 761: Steel *ab-initio*, 3rd funding period granted by DFG; newly founded Max Planck Fellow Group on "Self-Reporting Materials" by J. Schneider) and Ruhr-Universität



Bochum (SFB/TR 103: Next Generation Single Crystalline Superalloys, 2nd funding period granted by DFG; International Max Planck Research School (IMPRS SurMat) which is managed by E. Gattermann and A. Erbe; Center for Electrochemical Sciences; external research group of the MPIE on “High Temperature Materials” by G. Eggeler). The IMPRS SurMat has been recently successfully evaluated and prolonged, extending the number of members to now also including colleagues from the University Essen-Duisburg.

Recently several group leaders of MPIE received professorship offers from prestigious universities such as from Ohio State University, University Bern, Erlangen University, RWTH Aachen University, Massachusetts Institute of Technology MIT and the Korea Institute of Science and Technology KIST.

MPIE members are also involved in several science community and service activities such as serving as vice-president and president of the Max Planck Society (M. Stratmann), as member of the reviewer panel (Fachkollegiat) of the German Research Foundation DFG (J. Neugebauer) and as member of the German Council of Science and Humanities (Wissenschaftsrat) and the Joint Strategy Commission (Strategiekommission) for the German Excellence Initiative (D. Raabe).

Several highly visible awards were won by MPIE members in the past years (end 2012 - 2015), among them an ERC Starting Grant, 4 Alexander von Humboldt Awards for Senior Professors, the Otto-Hahn Award, the NRW Innovation Award, the DECHEMA-Award and 2 BMBF Junior Research Group Awards to name but a few important recognitions.

In order to stay connected to graduates and former scientists of the Institute and to benefit from their experience MPIE established a corresponding alumni network.

A new website has been launched in spring 2015 presenting the Institute’s research and achievements to different target groups ranging from scientists over students, journalists, politicians, industry representatives and school students to the wider public community. Furthermore a new series of lectures named “KopfSalat” has been started in autumn 2014 to increase the visibility of the Institute within the region. In this series high ranking scientists present their work to a broad regional public audience.

Another important transition currently affects the Institute’s future institutional core funding and legal status: The MPIE is one of the longest existing Max Planck institutes since its establishment in 1917 as Kaiser-Wilhelm-Institut für Eisenforschung. In 1971 it was rendered into a company with GmbH legal form and basic budget sharing between the Max Planck Society and the German Iron and Steel Institute VDEh of 50% each. Effective as of 2016 it is intended that the institutional VDEh contribution will be stepwise reduced over a period of three years to arrive at an overall smaller permanent budget fraction. The corresponding negotiations on alternative funding and temporary cost reduction options are currently ongoing. This shift in the institutional and legal setting will be used by MPIE to sharpen and shape its scientific focus along its mission of understanding and designing complex nanostructured materials under real environmental conditions down to the atomic scale.



Independent Research Groups

Independent Max Planck Research Group on Nanoanalytics and Interfaces

Group Head: C. Scheu

Research Mission and Scientific Concepts

The independent research group “Nanoanalytics and Interfaces (NG)” (Fig. 1) was established in April 2014 and is headed by Christina Scheu who holds on the same time a full professorship at the RWTH Aachen. The main research field of the NG is the in-depth characterization of novel nanostructured materials and interfaces via electron microscopy techniques. State-of-the-art transmission electron microscopy (TEM) is employed to obtain insight into the atomic arrangement, chemical composition and bonding behaviour down to the atomic scale. The results are used to establish e.g. growth



Fig. 1: The NG group at the IAMNano workshop in Hamburg (summer 2015).

models for nanostructures and structure-properties relationships. In addition, strategies are developed to improve the properties and stability of the materials of interest. The investigated materials systems are ranging from oxides, nitrides, carbides to polymers and composites with applications in photovoltaics, fuel cells and electrochemical cells. Furthermore, thin films used for metal contacts and protective layers are studied. In part, the nanostructures and thin films are grown in our group via wet chemical synthesis or physical vapour deposition routes. In addition, we are in close cooperation with project partners developing novel materials and several of our projects are funded by third parties such as the DFG including a partial project within the SFB 761/3 “Stahl *ab initio*” (see p. 52).

To elucidate the relationship between structure, synthesis and properties of nanostructures, we

use various TEM techniques. High-resolution TEM (HRTEM) and high-angle annular dark-field (HAADF) scanning TEM (STEM) are applied to obtain information on the local arrangement of atoms and to study defects with a resolution down to the atomic scale [1, 2]. The three-dimensional morphology of nanostructures is investigated by bright-field and dark-field imaging as well as electron tomography (the latter in close cooperation with international partners [3, 4]). The chemical composition of novel materials [5, 6], individual nanostructures [2, 7, 8] and interfaces [9, 10] are analyzed by energy-dispersive X-ray spectroscopy (EDX) and electron energy-loss spectroscopy (EELS). The EELS measurements enable a localized investigation of the electronic structure and optical properties (e.g. band gap [2, 11, 12]). The bonding behaviour and oxidation state of various nanostructures is determined by analyzing the electron energy-loss near-edge structure (ELNES) of individual, element-specific edges [6, 7]. For a detailed interpretation of EELS data, *ab-initio* calculations [6, 11] or a comparison with well-defined standard materials [7] are employed. *In-situ* TEM heating experiments are currently conducted to investigate phase transformations and structural changes on an atomic scale, similar as in an early work where we discovered the *in-situ* growth of alumina nanowires [13].

The environmental pollution and the climate change on the one hand and the increasing demand of energy supply together with decreasing gas and coal resources on the other hand have intensified the research on alternative, renewable energy sources. New concepts are developed for producing and storing energy as well as fuel - like hydrogen - in an environmentally friendly way and ideally at low costs. Besides novel materials and material combinations, the morphology plays a key role. Nanostructured materials offer the advantage of a large surface area/interface area to volume ratio. This leads to more efficient light harvesting devices, which are used to convert solar energy in either chemical (water-splitting) or electric energy (solar cells). Furthermore, these nanostructures can be often synthesized in an environmentally friendly way using fast and simple synthesis strategies. The NG group has several research activities in the field of nanostructured materials for renewable energy sources as outlined in the following.

Nanostructured Materials for Photovoltaics and Photoelectrochemistry

Nanostructured, non-silicon based thin-film photovoltaic devices are potential candidates for low-cost electricity generation. In one of our research projects we synthesize CuInS_2 thin films and nanostructures via a solvothermal route developed earlier [14,15]. With this approach crystalline solid materials can be grown at low temperatures and in the case of CuInS_2 in the Chalcopyrite modification at 150 – 160 °C. Nanostructured CuInS_2 thin films (Fig. 2) show a strong absorption behaviour over the whole visible light spectrum, possess a direct band gap of about 1.5 eV like the bulk phase and can act as both electron or hole conductor depending on the chemical composition and we will . The chemical composition will be analysed in more detail by EELS, EDX and atom probe investigations together with the department Microstructure Physics and Alloy Design (MA). In addition, we plan to perform electrochemical tests with the department of Interface Chemistry and Surface Engineering (GO), since CuInS_2 is reported to work as a photo-catalyst for light-induced splitting of water to produce hydrogen fuel, which is a central research topic of the NG (see also below).

Another research example is based on regular arrays of n-type large band gap semiconducting metal-oxide nanowires (e.g. TiO_2). These networks can be used as electrode materials in e.g. water-splitting devices or excitonic solar cells like dye-sensitized or hybrid solar cells. In the case of hybrid solar cells, the nanowire network is filled with an organic p-type semiconductor (e.g. poly(3-hexylthiophene) (P3HT)) which acts as absorber as well as a hole conductor. Due to the large interface, an efficient charge separation and thus a high efficiency is expected. However, the nanostructured photovoltaic devices lack behind the expectations. The factors limiting the performance were evaluated together with Prof. Lukas Schmidt-Mende from the University of Konstanz in project funded by the German Research Foundation (DFG). We have explored the atomic arrangement, crystal modification, band gap, and chemistry of hydrothermally grown nanowires on the atomic scale using various TEM techniques. The obtained information was correlated to the synthesis conditions and based on these results a growth model was developed which allows to explain the observed defect structures [1]. The TiO_2 nanowires crystallize in the rutile modification and exhibit faceted morphology with the {110} surface dominating. The growth is parallel to the <001> direction with a truncated growth front formed by {111} and {001} facets. The nanowires have a single crystalline bottom, but with ongoing crystal growth planar defects such as stacking faults occur, which form the starting point of a defect cascade causing

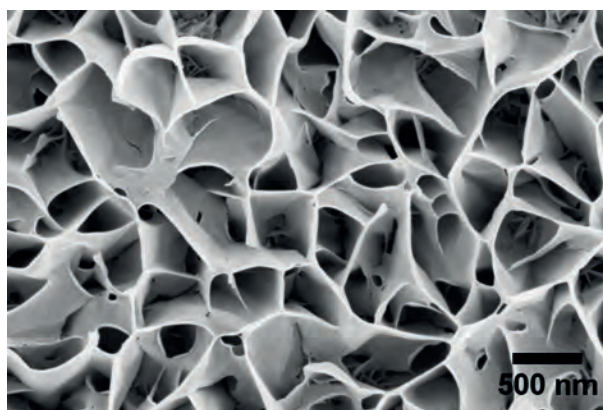


Fig. 2: SEM image of a nanostructured CuInS_2 thin film synthesized by a solvothermal method in a teflon-lined stainless steel autoclave at low temperatures. The synthesis was done following a protocol published in [14,15].

a finger-like structuring of the nanowire top (Fig. 3). This is most likely caused by steric hindrance and electrostatic repulsion at the {001} facets [1]. The occurring free surfaces as well as the stacking faults within the interior of the nanowire hinder an efficient electron transport and thus are responsible for the low efficiency of these solar cells [1, 16]. Ex-situ heating experiments performed at elevated temperatures revealed that the fingers at the nanowire top and the planar defects can be removed, but voids are formed within the nanowire interior. However, when applied in nanostructured solar cells, these voids have comparable little effect on the transport properties compared to the fingers [16]. A pronounced improvement of the cells was also achieved by creating nanowires with a highly conductive core via Sn^{4+} doping and a TiO_2 anatase shell which leads to a reduced charge carrier recombination [17]. Inspired by the promising results, we plan to do *in-situ* TEM heating experiments to uncover the structural changes occurring during the thermal treatment. In addition, a detailed study of the void

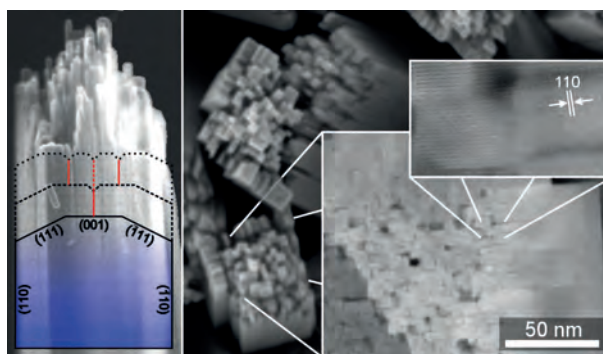


Fig. 3: Secondary electron STEM image (left), top-view SEM image (middle), cross-section STEM images (right) of a hydrothermally grown TiO_2 nanowire. The nanowire has a faceted morphology with individual fingers at the top. Figure taken from [1]. The (110) lattice spacing in TiO_2 is 0.32 nm.



shape within the nanowires by electron tomography is planned, in order to develop models of how they form and to detect possible oxidation state changes of the Ti ions at the surface/defects using EELS. The equilibrium shape as well as the surface structure will be analyzed in cooperation with the department of Computational Materials Design (CM). To further reduce the recombination, e.g. Nb_2O_5 core shell structures will be fabricated.

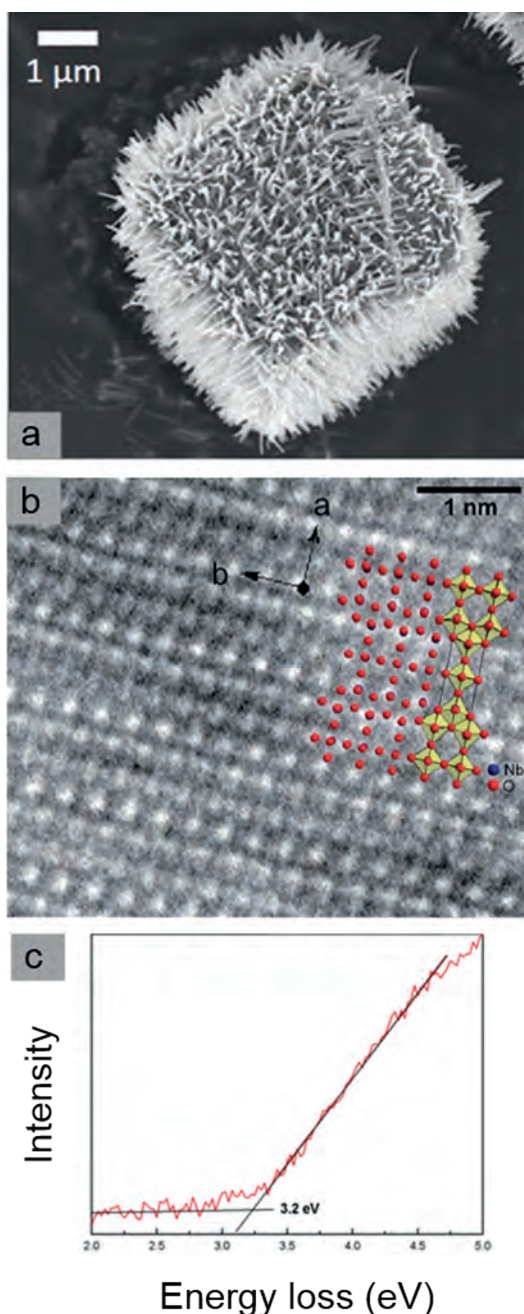


Fig. 4: SEM image (a) of a $\text{Nb}_3\text{O}_7(\text{OH})$ cube grown hydrothermally without a template consisting of a network of individual nanowires. (b) STEM image of a nanowire with indicated Nb and O positions in blue and red, respectively and (c) low loss spectrum to determine locally the land band gap. All images taken from [2].

In general, Nb_2O_5 as well as $\text{Nb}_3\text{O}_7(\text{OH})$ compounds can be considered as alternative materials for replacing TiO_2 . They possess a high chemical stability and a similar band gap of about 3.1-3.2 eV like TiO_2 . Besides the band gap, the energetic position of the upper valence and lower conduction band level is important for application as electrode material or electro-catalyst in energy and fuel generating devices. First experiments indicate suitable band positions for both compounds. We developed a one-step, template-free hydrothermal synthesis approach which allows us to grow crystalline $\text{Nb}_3\text{O}_7(\text{OH})$ superstructures at low temperatures of 200°C [2]. The superstructures form cubes which consist of hierarchically ordered 3D nanowire networks (Fig. 4). X-ray- and electron diffraction analyses revealed that the individual nanowires forming the 3D network are single crystalline $\text{Nb}_3\text{O}_7(\text{OH})$ with $\langle 010 \rangle$ growth direction (Fig. 4). The width of the nanowires is ranging from 10 to 50 nm and can be controlled by the synthesis conditions. The band gap determined globally using UV-Vis and locally in the TEM using low loss EELS revealed a value of 3.2 eV. The analysis of the ELNES of the Nb- $\text{N}_{2,3}$ and O-K edge indicate a nominal oxidation state of +5 of the Nb ions [2]. First photo-catalytic experiments conducted by our project partner Prof. Bettina Lotsch (MPI for Solid State Research, Stuttgart, and Ludwig-Maximilians Universität (LMU), Munich) and her team indicate that they are promising electro-catalyst for producing hydrogen fuel. In addition, the mechanical stability of the $\text{Nb}_3\text{O}_7(\text{OH})$ cubes and individual nanowires will be investigated in close cooperation with the department of Structure and Nano-/Micromechanics of Materials (SN). This information enables us to estimate how we can use them e.g. as paste for different applications while maintaining their electronic properties.

The NG also participates in the DFG priority programme 1613 “Fuels Produced Regeneratively Through Light-Driven Water Splitting: Clarification of the Elemental Processes Involved and Prospects for Implementation in Technological Concepts”. Together with our project partners we strive to develop novel metal oxide photoabsorbers and synthesize them in different nanomorphologies to achieve efficient electrochemical and photoelectrochemical water splitting. The selection of viable material candidates is assisted by the group of Prof. Rossitza Pentcheva (University of Duisburg-Essen) via density-functional theory (DFT) calculations, synthesis and photoelectrochemical characterization are performed by the groups of Prof. Thomas Bein and Prof. Dina Fattakhova-Rohlfing (both LMU Munich) and we contribute by in-depth TEM and focused-ion beam (FIB) studies. In the first funding period, we focused heavily on improving hematite (Fe_2O_3) as a photoanode material. In an initial experiment we optimized a nanostructured hematite absorber layer

by Sn-doping [18] and found a strong increase in the rate constant for water oxidation. TEM investigations revealed an enrichment of Sn at the surface of the individual Fe_2O_3 nanoparticles, which is most likely responsible for improving the catalysis taking place at the surface. For the first time, this study showed that selective surface doping can have a catalytic effect in hematite photoabsorbers. We also worked on developing hierarchical systems consisting of an absorber layer and a highly conductive macroporous scaffold. Synthesis strategies were developed to fabricate novel macroporous transparent conducting oxide (TCO) scaffolds with excellent electron transport properties [19]. The latter is related to the formation of a continuous crystalline network, which was investigated by TEM. Due to the insight obtained during these studies, we successfully designed effective hierarchical systems consisting of a macroporous, conducting TCO scaffold (antimony-doped tin oxide) infiltrated with a Sn-doped Fe_2O_3 absorber layer [20]. Another strategy is to improve catalysis by ultrasmall Co_3O_4 nanocrystals which are homogeneously distributed on the surface of the mesoporous Fe_2O_3 absorber layers [21].

Materials for Fuel Cells

Besides materials for photovoltaic devices and (photo)-electrochemical cells, we are also involved in material development and understanding of degradation processes in fuel cells. These research activities are done in close cooperation with our industry partner and are funded by the BMWi (Federal Ministry of Economics and Technology). Fuel cells convert chemical energy of a fuel into electrical energy via catalyzed redox-reactions on electrodes. They are environmentally friendly as during operation only water and heat are produced and a high efficiency is typically achieved which outperforms conventional power plants. However, fuel cells have the disadvantage of high production cost mostly due to the use of expensive catalyst materials and degradation phenomena which limit their lifetime.

We are working on high-temperature polymer electrolyte membrane fuel cells (HT-PEMFCs), where a polybenzimidazole (PBI)-based membrane is acting as proton conducting electrolyte and separating the adjacent anode and cathode from each other. The aim is to establish an understanding of excellent materials performance coupled with a high stability and low degradation rate. To achieve that goal, we analyze the microstructure of the different components before and after operation in detail to uncover the processes occurring during degradation. With this information at hand, we modify individual components as well as the whole fuel cell stack. In the case of the PBI-based membrane we found that longer thermal post-curing times lead to reduced degradation due to a change in the ordering of the polymer chains [22]. We also investigated the effect of adding silica nanoparticles to the membrane and observed an increase in stability due to the presence of the nanoparticles [23]. Detailed EDS and EELS studies of membranes containing differently sized nanoparticles revealed that they are homogeneously distributed and consist of amorphous silica [24]. In experiments together with Prof. Viktor Hacker (Graz University of Technology), we investigated the long-term behaviour of platinum cobalt catalysts and found that they have a similar performance like the more expensive pure platinum catalysts [25]. We also replaced the anode carbon support material by tungsten oxide and investigated the performance and degradation behaviour after different operation times and modes including start-stop-cycles [26]. We found three dimensional networks of few nanometre thin, crystalline Pt rods on the WO_{3-x} grains with a size of up to about $1 \mu\text{m}$ after the deposition (Fig. 5). These rods decrease in size during operation and Pt is diffusing into different areas of the fuel cells, including the membrane [26]. Nevertheless, the degradation rate of the fuel cells containing the WO_{3-x} based anode was found to be lower compared to fuel cells using the standard carbon based anodes. In future experiments, we plan to study different noble metals

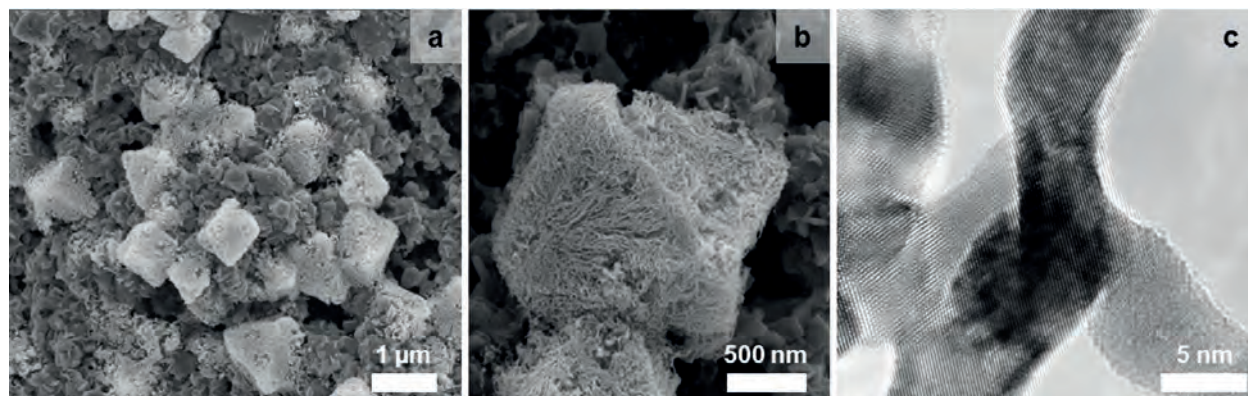


Fig. 5: SEM images (a,b) of Pt networks on WO_{3-x} anode support materials. The network consist of Pt nanorods with a few nanometer in diameter. The deposition of Pt is described in detail in [26].



and alloys as catalyst, and different high surface area carbon materials as well as various transition metal oxides as catalyst support material. These studies will be in close collaboration with the GO. In addition, we want to understand the growth of the Pt networks on the WO_{3-x} grains and will investigate different growth regimes.

Thin Films

As outlined, many of the nanostructures we investigate are prepared by wet-chemical synthesis protocols. Another approach we are working on is nanostructuring induced by controlled solid-state dewetting of thin metallic films. This project is done in close collaboration with the SN. In a first step, we grow epitaxial metallic Al films on single-crystalline Al_2O_3 substrates using molecular beam epitaxy and then perform annealing experiments using different

conditions by varying the atmosphere, dwell times and annealing temperatures. FIB sectioning and TEM studies revealed that drum-like voids covered by a thin oxide layer which form during solid-state dewetting. Although the experiments were performed at only 600°C , single crystalline sapphire ridges form at the Al/sapphire interface (Fig. 6). The thickness of the Al film increases locally in this region of the sapphire ridge compared to the original film thickness. The forces involved in the dewetting process will be evaluated in collaboration with the CM. Future studies will concentrate on the determination of the grain boundary structure, since preliminary experiments indicate that specific grain boundaries are acting as nucleation sites of dewetting. Also here, a close link to the CM is given, since DFT calculations provide a detailed understanding of grain boundary structure and energy. The thermal stability and dewetting studies are also relevant for thin films in general, since this causes major problems for applications as conductive lines or protective coatings. Together with the SN and the newly established Max Planck Fellow group of Prof. Jochen Schneider (see p. 21) we investigate thin Mo_2BC thin films on Si substrates with the aim to understand the microstructural evolution during annealing. As grown, these films are amorphous and are then transformed to a nanocrystalline state when thermally treated. Preliminary experiments conducted with the GO demonstrated that they can also be used as electrodes in water-splitting devices.

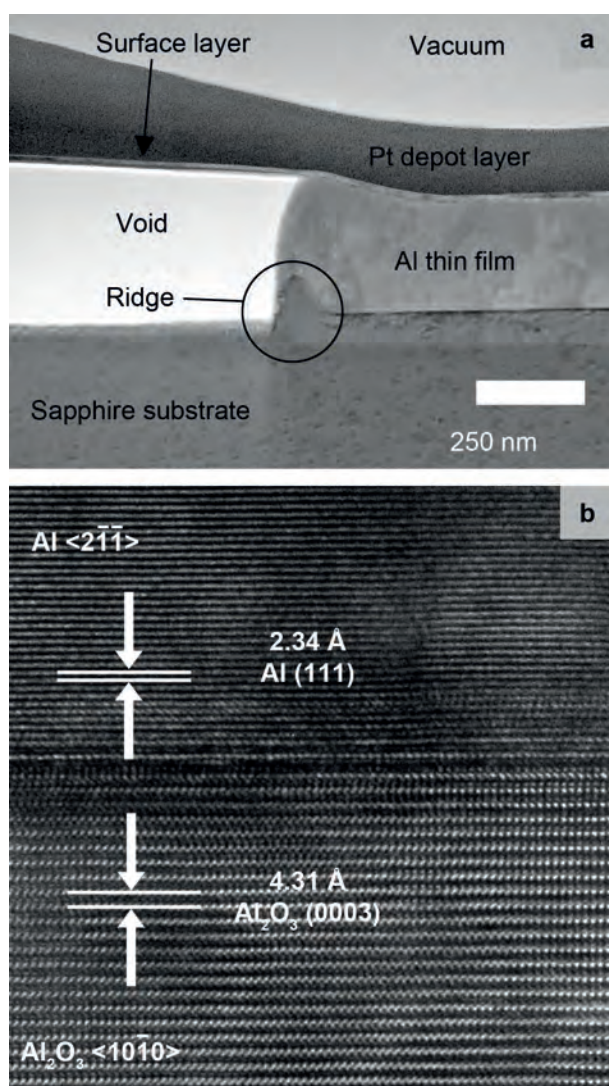


Fig. 6: (a) STEM image of a FIB cross-section showing the formation of a drum-like void and ridge after solid-state dewetting of a thin epitaxial Al film on a single crystalline Al_2O_3 substrate. (b) HRTEM micrograph of the Al/ Al_2O_3 interface relationship in (b) indicating a low-index orientation relationship.

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Research Projects in Progress

Betzler, Scheu (in collaboration with B. Lotsch*, L. Schmidt-Mende**, J. Ciston***, U. Dahmen*** (*LMU Munich and MPI for Solid State Research, **University of Konstanz, ***National Center for Electron Microscopy, USA)): In-depth investigation of novel 3D niobium oxide superstructures

Folger, Scheu (in collaboration with A. Wisnet*, A. Hartschuh*, L. Schmidt-Mende** (*LMU Munich, **University of Konstanz)): Synthesis and characterization of TiO₂ nanowires for electrochemical applications and solar cells

Frank, Scheu: Chalcopyrite nanomaterials for renewable energy applications

Gleich, Scheu (in collaboration with G. Dehm*, S. Djaziri*, J. Schneider** (*SN, **RWTH Aachen)): Characterization of nanolaminated materials by electron microscopy

Hengge, Scheu (in collaboration with Elcore, Munich): Structural and spectroscopic investigation of polymer based fuel cells

Hieke, Scheu (in collaboration in collaboration with G. Dehm, SN): From thin films to nanostructures

Müller, Scheu (in collaboration with T. Bein*, D. Fattakhova-Rohlfing*, I. Kondofersky*, J. Feckl*, A. Hufnagel*, R. Pentcheva** (*LMU Munich, **University of Duisburg): Investigation of photo-electrochemical water splitting-active nanostructures

Scheu (in collaboration with J. Mayer* (*RWTH Aachen)): Microstructure characterization – local structure, defect and chemical analysis (SFB 761/3 partial project C01)

Scheu (in collaboration with R. Hoffmann*, G. Langer**, C. Kirchlechner***, N. J. Balila*** (*LMU Munich, **Cambridge University; *** SN)): Morphology of *Emiliania huxleyi* coccospheres and effect on mechanical properties

Scheu (in collaboration with D. Raabe*, O. Cojocar-Miredin** (*MA, **RWTH Aachen): Microstructural and spectroscopic analysis of thermoelectrics



Max Planck Fellow Group on Self-Reporting Materials

Group Head: J. M. Schneider

**MATERIALS
CHEMISTRY**

**RWTHAACHEN
UNIVERSITY**

Future technology challenges will no longer be simply addressed by today's material and processing solutions, which are often based on trial and error. Instead guidance will be attained from correlative experimental and theoretical research bridging all length scales. It is e.g. evident that future engineering components will be made of materials that are self reporting. Self-reporting materials "communicate" damage of the material via changes of properties that can be measured in service as a consequence of chemical changes on the atomic length scale and/or phase transformations. Harnessing this would provide invaluable functionality for damage assessment and control and would constitute a major leap forward as the "health" of any material component exposed to mechanical loads could be monitored in service. This fascinating basic materials science with tremendous technological implications is the focus of Schneider's Max Planck Fellow Group. Established in October 2015, the group will synthesize and characterize charge density modulated materials (multilayers & nanolaminates) regarding their elastic and plastic

properties as well as their thermal and chemical stability. Based on these investigations and guidance from quantum mechanical calculations the charge density modulated materials with self reporting capabilities will be designed by substitution and/or addition of elements.

Thin film architectures with modulated charge density distributions will be grown by combinatorial vapour phase condensation as depicted in Fig. 1 (right). In a collaborative effort between the department Microstructure Physics and Alloy Design (MA) and Schneider it was demonstrated that the in Aachen grown multi-layered thin film architectures, shown in Fig. 1 (left) serve as model systems for the investigation of plasticity [1].

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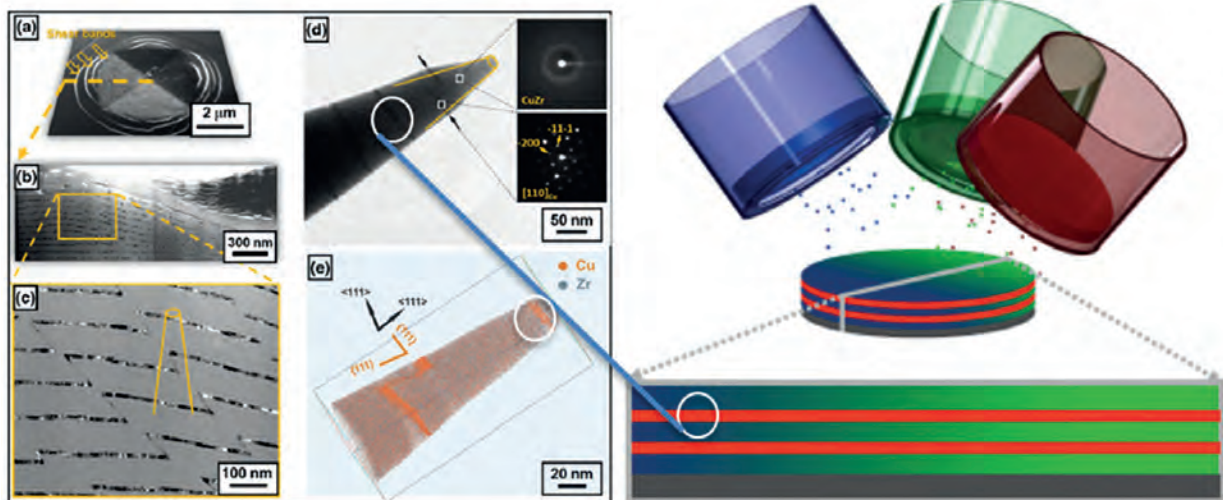


Fig. 1: Illustration of the combinatorial synthesis approach to be employed in the Fellow group for the multilayers and nanolaminates (right). The colour gradient indicates a gradient in chemical composition [1]. On the left the images a-e show a plastically deformed crystalline-amorphous nanolaminate. Similar coating architectures will be utilized as initial platform to study the fundamentals of self-reporting materials.



New Research Groups

Nanotribology

Group Head: S. Brinckmann

Department of Structure and Nano-/ Micromechanics of Materials (SN)

The mission of the group, which was established in December 2014, is to promote the understanding of tribology-formed microstructures including mechanical and chemical dominated layers and deformation on the nanometer scale. Initially, the focus is laid on the mechanical properties: plasticity, crack formation and growth, and the friction coefficient. We will study the dependence of the friction coefficient on the grain orientation and on secondary phases as well as on lubrication. Moreover, the tribology-induced microstructure evolution, i.e. grain refinement, grain re-orientation, as shown in Fig. 1, is a central theme of the group.

We will concentrate on the deformation of and due to microasperities, which resemble microstructural roughness that exists even on polished surfaces. Thereby, the research group will bridge the gap between conventional and atomistic tribology. Conventional tribology on the millimeter scale uses tribometers (e.g. pin-on-disk, disc-on-disc) to study friction & wear. As such, conventional tribology mimics the tribological deformation on the macroscale and includes all tribological mechanisms that interact with each other. Atomistic tribology uses the atom force microscopy (AFM) to scratch and image the surface, which resembles the deformation due to nanometer atomistic asperities. This atomistic roughness exists on any surface and leads to predominantly elastic deformation. By using the influence of the atomistic scale and focusing on the tribology of the nanometer scale, we can study fundamentally the individual mechanisms that occur on the macroscale.

A central benefit of the nanotribology is the ability to perform high throughput experiments (Fig. 2) using the same asperity, the same metal phase, the same lubricant and varying the friction power density, i.e. normal force and scratch velocity, over five orders of magnitude. Moreover, by varying the counter asperity radius and/or material, only small domains are required. Post-deformation inspection by AFM

and scanning electron microscopy (SEM) allows us to quantify surface roughness, local grain re-orientation, grain sizes, cracks (Fig. 1) and allows determining the influence of the friction power density.

In the future, the investigation of the chemical mechanisms of tribology will move more into the focus. We have constructed an electro-chemical cell for the nanoindenter that allows studying hydrogen embrittlement (see p. 31). In engineering applications, the outer surface layers are dominated by chemical processes, which result in a 10 to 100 nm thick

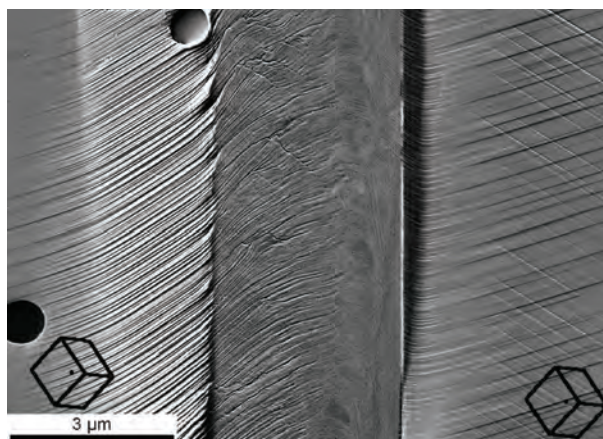


Fig. 1: Surface steps on the sides of a scratch that runs through a large grain of austenite. Although scratching is a symmetric loading and both sides have the same crystal orientation, the plastic deformation and the surface steps are non-symmetric. On the right-hand side, two slip systems have the same activity, while on the left-hand side one slip system dominates plasticity.

layer of sulfides, oxides and phosphates. If these outer surface layers are optimal, they result in ultra-mild wear rates, which are not measurable during state-of-the-art macroscale tribometer experiments. The nanotribology allows to separate the tribology mechanisms and to study the creation and growth of the surface layers individually and localized.

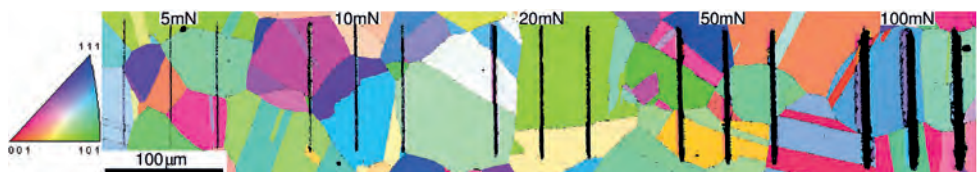


Fig. 2: Electron-Backscatter-Diffraction (EBSD) image of the scratched area. The normal direction grain orientation is encoded as shown on the left-hand side. The scratch direction is shown on the right hand side. The local grain orientation in the scratch proximity is disturbed by the deformation.



Interface Design in Solar Cells

Group Head: O. Cojocaru-Mirédin

Department of Microstructure Physics and Alloy Design (MA)

This new research group was established in 2013 with the support of the 'Bundesministerium für Bildung und Forschung' (BMBF NanoMatFutur competition). The mission of this group is to optimize solar cell efficiency based on an improved understanding of the relationships between chemical, structural, and electrical properties at the atomic-scale.

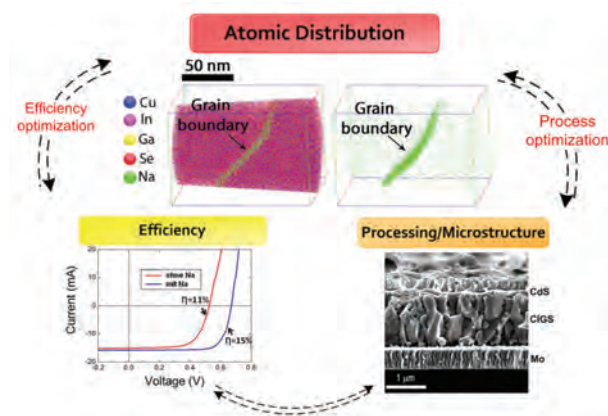


Fig. 1: The correlation between Processing/Microstructure, Efficiency and Atomic Distribution for a $\text{Cu}(\text{In,Ga})\text{Se}_2$ thin film solar cell.

During the past decade, designing new and beneficial internal interfaces in such photovoltaic materials has become crucial for controlling solar cell efficiency. Further improvements depend on a better understanding of the underlying atomic-scale interface mechanisms. A suited approach lies in mapping the cell's nanostructures by atom probe tomography for identifying the elemental distribution

and the associated interface phenomena. Examples are impurity segregation at grain boundaries or p-n junctions as well as clustering and diffusion effects.

The research group concentrates on crystalline silicon [1] as well as on $\text{Cu}(\text{In,Ga})\text{Se}_2$ and $\text{Cu}_2\text{ZnSnSe}_4$ thin-film [2] solar cells owing to their relatively high efficiency. To further establish c-Si and thin-film technology in this field both, the reduction in production costs and the enhancement of solar cell efficiency are essential. The latter aspect depends strongly on the internal interfaces as they can affect the transport of the photogenerated charge carriers. Moreover, the internal interfaces, such as grain boundaries and p-n junctions, are very often decorated by impurities which can profoundly affect cell efficiency [3], [4]. Fig. 1 illustrates that the efficiency of a $\text{Cu}(\text{In,Ga})\text{Se}_2$ solar cell has been improved by almost 50% by merely adding minor amounts of Na (~ 0.1 at.%) to the absorber layer. Furthermore, Fig. 1 reveals that this increase in efficiency can be directly linked with Na segregation at the $\text{Cu}(\text{In,Ga})\text{Se}_2$ grain-boundaries.

Therefore, the aim of this research group is to perform chemical, structural, and electrical characterization of the internal interfaces in solar cells by using latest nanometrology approaches, such as atom probe tomography in conjunction with complementary techniques such as electron backscatter diffraction, transmission electron microscopy, cathodoluminescence, and electron-beam induced current. The overarching goal is then to synthesize novel and improved multicrystalline Si and $\text{Cu}(\text{In,Ga})\text{Se}_2$ or $\text{Cu}_2\text{ZnSnSe}_4$ thin-film solar cells in direct collaboration with several institutes at the forefront of solar innovation.

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TIME-BRIDGE: Time-scale Bridging Potentials for Realistic Molecular Dynamics Simulations



Group Head: B. Grabowski

Department of Computational Materials Design (CM)



The European Research Council (ERC) rewards the most prestigious and competitive scientific funding in Europe. The criterion for selection is scientific excellence. In 2015, Dr. B. Grabowski was awarded an 'ERC Starting Research Grant' for his innovative project proposal: TIME-BRIDGE: Time-scale bridging potentials for realistic molecular dynamics simulations.

The main objective of the recently established independent group entitled TIME-BRIDGE is to address the time scale problem encountered in molecular dynamics simulations. This problem originates in the fast thermal motion of atoms which happens on a femtosecond time scale. The processes determining relevant materials properties, such as dislocation motion, are instead taking place on significantly longer time scales, sometimes even in the range of seconds. Therefore, a gap of many orders of magnitude needs to be bridged.

Within TIME-BRIDGE, the key idea to solve the time scale problem is based on the pseudopotential concept. This concept is well known in the field of electronic structure theory where it helps to resolve the problem of highly localized core electrons. In

TIME-BRIDGE a pseudo-potential will be developed that projects out the fast thermal vibrations of atoms allowing therefore to capture the relevant processes on large time scales. A core ingredient will be the quasiharmonic dynamical matrix which provides analytic solutions to the motion of atoms over arbitrary time scales.

Processes that will be studied using the new methodology will for example include nanopillar compression tests (Fig. 1). Such tests provide highly valuable information about the behaviour and interaction of the fundamental objects of plastic deformation. The performed simulations will be supported by complementary experimental measurements which will allow to carefully evaluate the developed techniques.

The TIME-BRIDGE project has a horizon of five years. Over this period, the funding covers three post docs, and a senior scientist. Dr. Grabowski and two post docs will be working on the theoretical developments and applications. One post doc will be performing the experimental measurements under the supervision of Dr. Kirchlechner from the department of Structure and Nano-/Micromechanics of Materials.

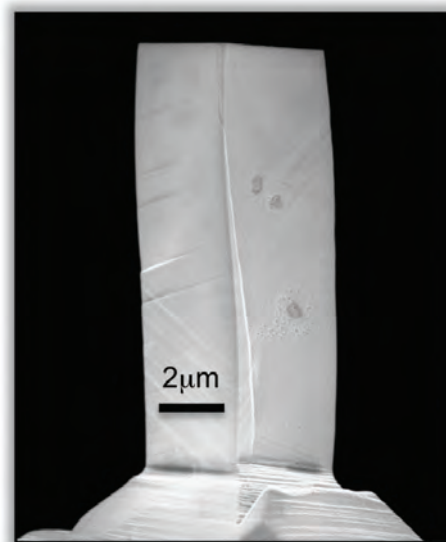
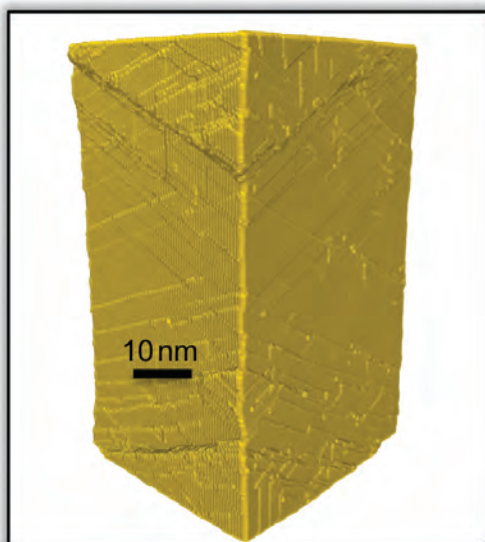


Fig. 1: The left figure shows a theoretical simulation of a nanopillar exposed to a compression leading to characteristic deformation lines on the surfaces. The right figure shows an analogous experiment monitored under a scanning electron microscope. So far the theoretical and experimental results cannot be fully matched. The developments within TIME-BRIDGE will provide the necessary tools and knowledge to tackle the challenge.



Alloys for Additive Manufacturing

Group Head: E. A. Jäggle

Department of Microstructure Physics and Alloy Design (MA)

In April 2015, a new non-permanent research group has been established working on alloys and alloy design for Additive Manufacturing.

Additive Manufacturing (AM) is a rapidly maturing technology capable of producing highly complex parts directly from a computer file and raw material powders. It is different from conventional manufacturing methods in that the parts are produced additively, layer by layer, from raw material (e.g. powder, wire, liquid) until the desired shape is reached, as opposed to subtractive, e.g. by milling away material from a block of metal. Currently, AM is being adopted in more and more industries, specifically in aerospace, automotive, energy and (bio-) medical branches, and is being applied to produce an increasing number of diverse and highly complex products. This may lead to a revolution in manufacturing: The disruptive potential of AM lies in its ability to manufacture customised products with individualisation, complexity and weight reduction for free.

The first focus of the research group is to work on one of the central questions of materials science: How does the manufacturing process, in this case Laser Additive Manufacturing (LAM), influence the micro- and nanostructures of the employed alloys and hence their final properties? The group will not focus on the additive manufacturing process as such, but rather on its consequences to the material. For example, the rapid cooling and cyclic re-heating experienced by a part produced by LAM has an influence on the solidification behaviour of the material, introduces unwanted residual stresses (which can even lead to failure of the material) and can also trigger solid-state phase transformations.

Ultimately, the goal is not only to understand the peculiarities of the LAM process and its influence on the materials, but also to optimize existing materials and to design new, tailored materials. Since the existing materials that are in use today in the LAM processes have originally been developed for other processing routes, it is to be expected that their composition is not the optimum one for LAM. For example, the hot cracking behaviour seen in Ni-base superalloys produced by SLM might be alleviated already by small changes in the alloy composition that influence the grain boundary liquid film during the last stages of solidification.

Design of completely new alloys for LAM is so far almost entirely missing in the research landscape. The established alloys currently in use do not exploit the enormous opportunities inherent in this technique at all, leaving a profound gap towards its further development. In the research project “Advanced Alloy and Process Design for Laser Additive Manufacturing” (grant awarded by the research alliance between the Fraunhofer Society and the Max Planck Society), the project partners from MPIE and the Fraunhofer Institute for Laser Technology (ILT) are collaborating to develop tailor made alloys exploiting the unique characteristics of the LAM processes in combination with an adaptation of process engineering and process parameters to achieve alloy composition and properties *in-situ*. This can only be achieved by combining the expertise of the partnering institutes in the fields of alloy design and characterisation (MPIE) as well as process design and engineering (ILT).

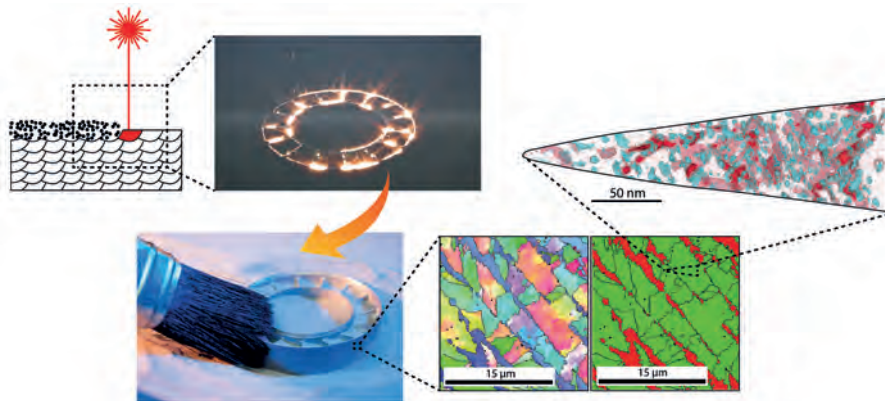


Fig. 1: Schematic depiction of the layer-by-layer build up of a part by a Laser Additive Manufacturing process (specifically: Selective Laser Melting), together with Electron Backscatter Diffraction phase map (green: ferrite, red: austenite) and orientation map showing the microstructure induced by solidification and an Atom Probe Tomography map showing the nano-structure induced by heat treatment (light/dark red: 10% / 24% Mo isoconcentration surface, teal: Ti/Mo=1 isoatomic-ratio surface). The material under consideration is 18Ni-300, a maraging steel (1.2709).

Nano-/Micromechanics of Materials

Group Head: C. Kirchlechner

Department of Structure and Nano-/ Micromechanics of Materials (SN)

Designing future's materials requires detailed knowledge of the fundamental mechanical properties across all length scales. But during macroscopic testing – i.e. in bulk samples – important details and mechanisms at the microstructural length scale remain unknown as they are being blurred by the collective behaviour and interplay in a complex microstructure. Recent advances in producing and deforming miniaturized mechanical samples, site-specific with yet unreached small dimensions, bring the former vision of closely following materials behaviour at the outmost important length scales into reality.

The new research group Nano- and Micromechanics of Materials, which started in March 2013, is probing the mechanical properties regarding plasticity, fracture and fatigue at the micron and sub-micron scale. But the spirit of nano- and micromechanics surpasses being a mechanical microscope by far: A central aspect of the group is to interlink the mechanical response to the underlying and evolving defect structure, i.e. to measure and observe dislocation densities and interaction mechanisms, crack length, phase transformations and change of electrical properties *in situ* during mechanical loading. This is accomplished by advanced microstructural characterization tools in-house (e.g. transmission electron microscopy) or at a large-scale synchrotron facility (e.g. ESRF, BESSY II). The final goal of the group is to develop material models at the microstructural length scale serving as guidelines for materials development and input for materials simulations.

Two approaches are followed in our group: Thin film testing, where typically a rigid film is deposited

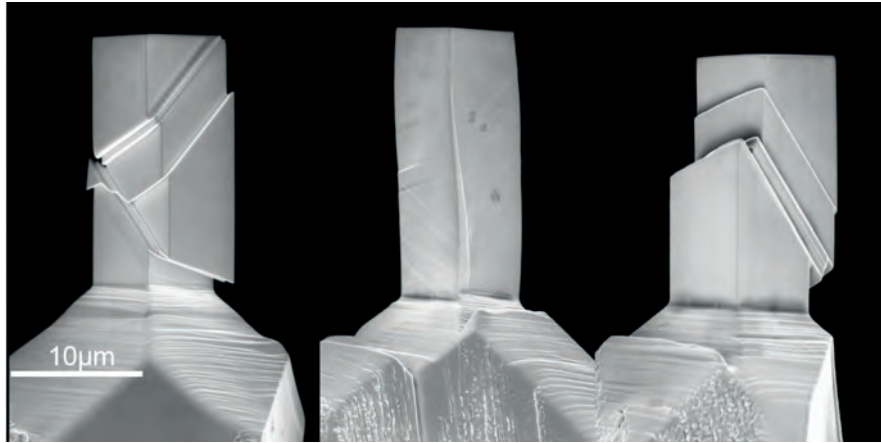


Fig. 1: Example of compressed copper micro pillars. The pillars in (a,c) are single crystalline with clear slip steps through the entire pillar documenting free glide of dislocations. The centered pillar (b) is comprised from the two single crystalline ones at the side, with a grain boundary in the center. Many more faint slip steps document the important role of grain boundaries as obstacles for dislocation motion.

on a compliant substrate and subsequently strained in a tensile device; and focused ion beam (FIB) based micromechanics, where test specimens being not too different from their large scale counterparts are produced in a FIB microscope and tested in a nano-indentation device (see “New Scientific Laboratories”).

Within the past years, the role of phase transformations on the adhesion and fracture properties of metastable face centered cubic cobalt thin films had been studied [1]. Furthermore, dislocation-grain boundary interaction mechanisms [2,3] as well as fatigue in pure copper in single crystals [4] and bicrystals had been investigated. The group hosts already 9 scientists and has established a strong network in Germany, Europe, the United States of America and Japan including several scientific industry collaborations.

In the future, high temperature capabilities will be built up to study e.g. brittle to ductile transition phenomena of engineering materials, high temperature properties of intermetallics and hard coatings.

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Advanced Transmission Electron Microscopy

Group Head: C. Liebscher

Department of Structure and Nano-/ Micromechanics of Materials (SN)

Mission: The mission of the Advanced Transmission Electron Microscopy group revolves around the characterization and design of structural and functional materials from the atomic level to mesoscopic length scales. The group was established in April 2015 to link atomic resolution transmission electron microscopy (TEM) with novel *in situ* TEM techniques. From a materials perspective, key aspects are the investigation and development of interface dominated materials and the related interface structures and properties, the interplay of local atomic arrangements and the corresponding deformation behaviour and the design of novel nanostructured materials.

Atomic scale characterization: A core theme of the group is the length-scale bridging characterization of materials to establish a complete microstructural concept providing the basis for new material development. An example of such an approach is given in the figure below, where the arrangements of precipitate structures and their evolution is characterized from a mesoscopic scale down to the atomic level [1]. This approach enables a direct link to theoretical predictions on the basis of first-principles calculations of phase stabilities and interfacial phenomena. Aberration-corrected TEM plays the key role in determining the chemistry, bonding characteristics and crystallography with sub-nanometer resolution. Such advanced techniques require the development of new imaging and data processing schemes representing a further area of research. The coupling of atomic resolution imaging with atomistic simulations will be

essential for modern and innovative material design and is therefore considered to be another core part of the group.

***In-situ* microscopy:** The understanding of material behaviour on the nanoscale through *in situ* microscopy techniques will complement the atomic resolution characterization. The focus lies on *in situ* deformation and heating methods, since atomic arrangements are dictating the mechanical and thermodynamic properties of materials. The necessity for decoding the material behaviour under externally applied conditions is to have a full picture and control of the nanostructure of a material [2]. A fundamental correlation of atomic scale characterization and *in-situ* deformation studies is extending the research topics of the Advanced TEM group. The atomistic structure of segregation effects at interfaces – such as grain boundaries – and the respective nanoscale deformation behaviour will serve as paradigm for this novel, comprehensive approach.

Current activities: The ongoing research activities are focusing – but are not limited – on the characterization of nanocrystalline, metastable Cu-Cr alloys [3] and the respective bonding characteristics, the atomic structure and segregation behaviour of interstitial and substitutional elements at grain boundaries, the design and characterization of nanostructured materials with hierarchical microstructure [1] and the microstructural investigation of semiconducting nanowires.

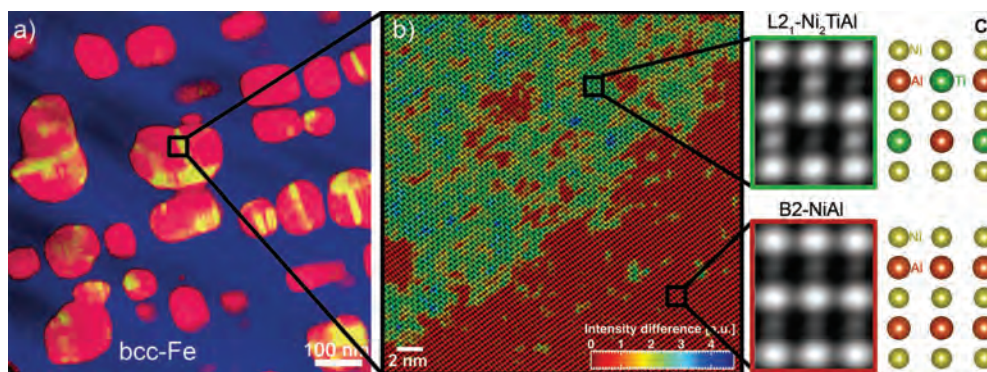


Fig. 1: a) Hierarchical microstructure of a precipitation strengthened ferritic alloy with two-phase B2-NiAl / L₂₁-Ni₂TiAl precipitates. b) Atomic resolution micrograph of a B2-L₂₁ interface. The colouring scheme is based on the local order parameter of the B2- and L₂₁-phases. c) Atomic arrangement of the B2-NiAl and L₂₁-Ni₂TiAl phase with a schematic projection of the corresponding crystal structures.

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Electrochemistry and Corrosion

Group Head: M. Todorova

Department of Computational Materials Design (CM)

The “Electrochemistry and Corrosion” group was established in October 2015 in the CM and is headed by Dr. Mira Todorova. The objective of the group is the development of quantum mechanics based multi-scale simulation techniques and their application to problems in electrochemistry, with a strong focus on corrosion. The activities in the group are tailored to provide in-depth insight into the functionality and evolution of materials properties under the influence of realistic environments. The thus gained understanding of experimental observations and the identification of relevant physical mechanisms will aid the development of strategies to counteract, retard or suppress degradation phenomena in materials.

The complexity of solid/liquid interfaces, which are at the core of most electrochemical processes of practical importance, presents a major challenge: all the obstacles faced in the context of the individual phases comprising such an interface come together and are further enhanced by the presence of spatially rapidly changing electric fields. For example, the statistical nature of liquids, translates into long sampling trajectories of molecular dynamic simulations. On the other hand, the nature of a solid electrode (metal or semiconductor/insulator), even in the most simple case where it can be described as a (defect free) model system, is of central importance in the context of the potential drop inherent to a solid/liquid interface, and usually translates in length scales which are beyond the extend of unit cells used in first-principles calculations. However, an accurate description of the electronic structure (of both solid

and liquid), including the alignment of band structure and redox levels, is essential for understanding the mechanisms driving the involved reactions. While this list is not complete, it illustrates that a direct brute force quantum mechanical approach is unfeasible and well controlled approximations and efficient numerical formulations are of utmost importance.

A first -step in this direction was the development of an approach which links *ab initio* calculations quite naturally to experimental observables characterising and determining the state of an electrochemical system, such as the pH-scale and the electrode potential. The approach exploits the similarities between charged defects in semiconductors and ions in liquid, is based on a fully grand-canonical description of both ions and electrons, and unifies and “translates” concepts in semiconductor defect chemistry and electro-chemistry [1]. This approach provides a sound basis for our further developments which, on one hand, strive to benchmark, analyse and quantify the required and achievable accuracy of presently available *ab initio* techniques focusing on the calculations of ion solvation and, on the other hand, aims to identify mechanisms driving reactions, for example, the construction of defect stability phase diagrams which enable, e.g. to identify the point defects which are relevant in the context of oxide film growth (see p. 161).

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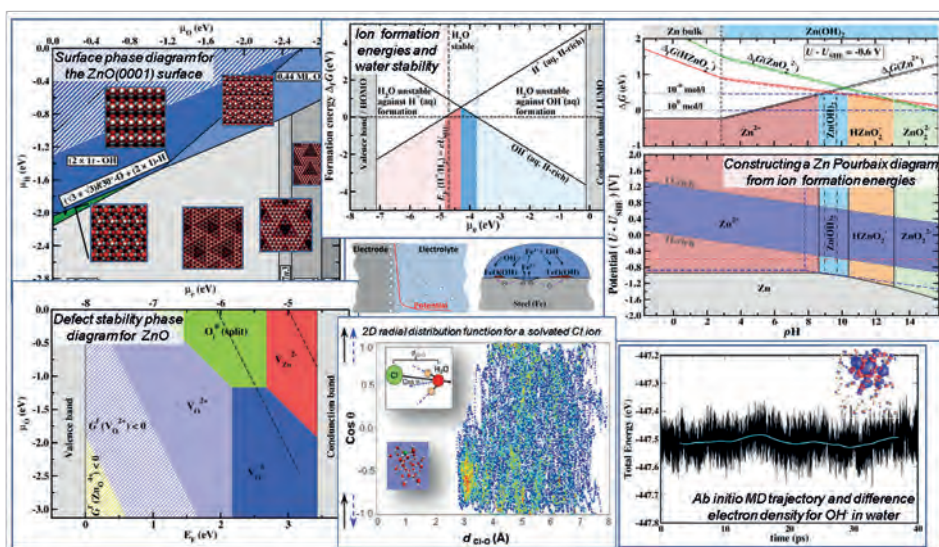


Fig. 1: Sketch of current activities in the group, which include surface, interface and bulk stability with respect to the environment and native defects (Pourbaix and phase diagrams) and ions in water (2d radial distribution function, molecular dynamics trajectory).



Atomistic Modelling

Group Head: S. Wippermann

Department of Interface Chemistry and Surface Engineering (GO)

This new *ab initio* theory group is located inside the otherwise exclusively experimental GO department and was established in February 2013. Additionally, in January 2014 the group leader Stefan Wippermann received a grant of 1.17 Mio. € from the German Federal Ministry for Education and Research (BMBF) within the NanoMatFutur competition to establish an independent junior research group for “Semiconducting Nanocomposites with Tailored Electronic and Optical Properties”. This independent group will be incorporated into the Atomistic Modelling Group. We work in direct collaboration with experiment to gain insights into key processes at solid-solid and solid-liquid interfaces, nanostructures and in the gas phase, which are inaccessible by other means. Employing predictive *ab initio* modelling techniques based on density functional theory and beyond [1], we focus on the general fields of materials for solar and chemical energy conversion and storage.

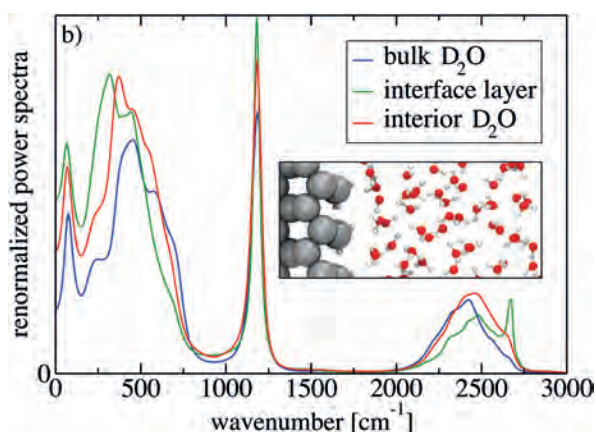


Fig. 1: Vibrational power spectra of the deuterated $\text{Si}(100)\text{-D}_2\text{O}$ interface, partitioned into contributions from bulk and interfacial water. A red-shift in the libration modes is observed compared to bulk water and a shoulder in the stretching modes of interfacial water, related to a breaking of the H-bond network.

One particular research area is the combination of atomistic thermodynamics and theoretical spectroscopy. Many of the surface characterization and analysis techniques common to surface science under ultra high vacuum conditions are not applicable to solid-liquid and solid-solid interfaces. Techniques to probe such interfaces, most notably spectroscopy, are often very indirect. We employ atomistic thermodynamics to identify and predict relevant structural motifs and processes at interfaces. Performing *ab initio* calculations of, e.g., vibrational, optical excitation and electron paramagnetic resonance (EPR) spectra allows for direct comparison to experiment.

Solid-liquid electrode-electrolyte interfaces are of tremendous technological importance in fields as varied as, e.g., electrolysis and -catalysis, corrosion, batteries, fuel cells and solar water splitting devices. Often multiple interfaces are involved between

metals, semiconducting metal-oxides and liquid water. In addition, ions solvated in the electrolyte screen the charge on the solid surface, forming an electrochemical double layer. A detailed understanding of such interfaces at the atomistic level has so far remained elusive. Significant challenges persist even in the *ab initio* description of the relevant bulk materials, e.g. transition metals and water. Therefore coupled theory-experimental investigations on well controlled and characterized systems and interfaces play a key role, in order to develop robust strategies to interpret experiments and validate theory. Starting from model interfaces, such as well-defined single-crystalline semiconductor-water interfaces, we investigate the atomistic and electronic structure of both the surface and the electrochemical double layer, thermodynamic stability, solvation of the surface, interaction with ions in the electrolyte, and the dependence on pH and the applied bias potential from first principles. One of our major scientific goals is to develop methods to predict the structure of electrochemical interfaces employing first principles atomistic thermodynamics, validate predicted structures against spectroscopic experiments and establish structure-property relationships.

Analogous considerations apply to complex nanometer-sized solid-solid interfaces, e.g. at surface-supported nanowires or nanocrystals embedded in a host matrix. Such nanocomposites are a promising approach to assemble functional materials with specifically designed electronic and optical properties, with potential applications as catalysts in electrochemistry, light absorbers for photovoltaics and light emitters in LEDs and lasers. Synthesis can be performed completely by inexpensive wet chemical solution processing. These nanomaterials may exhibit new functional properties not observed in conventional semiconductors, e.g. efficient carrier multiplication where an incoming photon creates more than one exciton [2,3]. The electronic and optical properties of these nanocomposites are dominated by interfaces and in particular defects at interfaces. We are systematically exploring the rich configurational space of technologically relevant nanocomposites. Subsequently, the obtained models are validated by means of joint theory-experimental IR, Raman and EPR spectroscopy. Such carefully developed models form an ideal basis to obtain key insights into their nanoscale physics and chemistry, allowing for a more rational device design.

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New Scientific Laboratories and Facilities

Micromechanics - Small Scale Testing Facilities

C. Kirchlechner

Department of Structure and Nano-/ Micromechanics of Materials (SN)

Probing material properties at the micron scale requires dedicated machines and setups for sample manufacture, sample testing, and *in situ* as well as post mortem defect analysis. Within the past three years capabilities to produce and deform micron and submicron sized samples have been built up in the department Structure and Nano- /Micromechanics of Materials and are briefly summarized here.

The largest share of small scale samples at the MPIE is nowadays produced via a focused ion beam (FIB) based route, for which a Zeiss Auriga® Crossbeam system was installed in 2013. The FIB is operated with 5 keV to 30 keV Ga⁺ ions focused typically to a beamsize in the range of some nanometres. In contrast to standard FIBs, the attached nano-patterning and visualization engine (NPVE, Fibics Inc, Ottawa, Canada) gives full access to the beam control. Thus, complex shapes with varying geometries requiring different dwell times locally can easily be set and manufactured in an automated way. By that, the NPVE helps us to surpass one of the bottle necks during micromechanical experiments: the manufacture of a statistically sound number of samples. Typical sample sizes produced via this route vary from several tens of nanometres up to 20 micrometres, which is in a size regime where a transition from a deterministic to a stochastic behaviour is observed, and therefore a high number of experiments is required.

On top of the dedicated sample manufacture, also testing rigs spanning over several orders of magnitude have been implemented in past years. Our indentation systems are either able to operate at a synchrotron beamline without obstructing the x-ray beam, are operated inside a scanning electron microscope (SEM), or in the transmission electron microscope (TEM). The variety of testable sample sizes ranges from some tens of nanometres (TEM, tested with Hysitron Pi 95 TEM Picoindenter®, Hysitron Inc., Minneapolis, USA) over several tens of micrometres (SEM, tested with ASMEC Unat II, Radeberg, Germany) to almost 500 μm (Laue diffraction, built at the MPIE). The various new loading rigs provide significant overlap in maximum force and strain rate and complement the existing

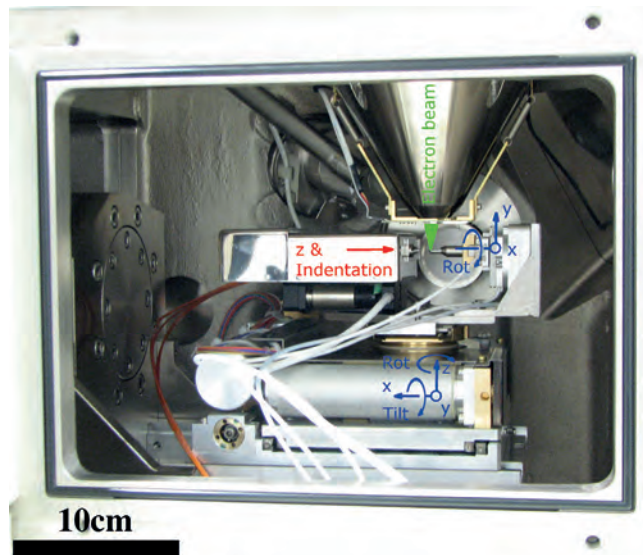


Fig. 1: Asmec Unat II installed in a Jeol JSM 6490 SEM, our standard testing rig for micron-sized samples. The counterparty (e.g. a diamond or tungsten tip) is marked with “Indentation”, with the sample support pin opposite of it. Sample and indenter can independently be moved along 4 axes with submicron resolution. In addition, the entire setup can be oriented with 5 additional axes, which allows for versatile experimental setups (Electron Backscatter Diffraction, ECCI).

(macroscopic) straining rigs at the MPIE. Thus, we are now able to probe material’s response from the low nanometre regime up to macroscopic sample and component dimensions, giving us the unique ability to understand the material’s behaviour across the relevant length scales.

All of the newly installed machines are aimed for *in situ* operation – i.e. advanced characterization tools as for instance the electron contrast channelling imaging (ECCI as pioneered in the Microstructure Physics and Alloy Design department) or dedicated environmental loading conditions using a hydroelectric cell (in collaboration with the Interface Chemistry and Surface Engineering department) – and thus, are well suited for answering advanced questions in various material systems.

To increase the throughput of our testing facilities with additional environmental capabilities (e.g. gases, temperature), a chamber equipped with an optical microscope (Olympus DSX 500i) is being built within the next year.



Nanoindentation and -tribology Laboratory

J. Duarte-Correa, S. Brinckmann

Department of Structure and Nano-/ Micromechanics of Materials (SN)

Tribology, i.e. friction and wear, depends significantly on five parameters: the material, the surface roughness, the counter material, lubricants and the environment. In engineering applications, boundary layers develop on the surfaces if the five parameters have been optimized and these layers consist of a chemically and a mechanically dominated zone. The former has a thickness on the order of tens of nanometer and the latter has a thickness on the order of micrometers. To reduce the friction, one has to reduce the adhesion of microasperities, which exist on any – even polished – surface. The study of adhesion, mechanical deformation, microstructure evolution, surface topology formation and the chemical processes surrounding tribology are at the center of the Nanoindentation and -tribology Laboratory (established June 2014).

In addition to the focus on tribology, the laboratory is set up to perform nanoindentation experiments, since both scientific research areas overlap. These nanoindentation experiments are either required for other research projects to extend the possibilities of the Institute or they set the basis of the deformation processes later extended to the lateral direction during tribology studies. Until recently, the nanoindentation experiments were limited to 10 mN at the Institute, which proved to be a limitation for very hard bulk materials and hard thin films. This limitation was especially detrimental for samples with a strong surface roughness.

The central equipment of this laboratory is the Keysight Nanoindenter G200 with a normal force range until 500 mN, which allows studying deformation even with blunt indentation tips. The normal force is applied with a coil-magnet assembly and the normal displacements are recorded by a capacitance gauge. In addition, the lateral forces in both directions are measured and the friction coefficient is evaluated. The transitional stage with nanometer lateral resolution allows targeting small phases during nanoindentation and micromechanical tests, and executing atomic force microscopy (AFM) like surface topology scanning prior and post deformation.

One key feature of the G200 is its ability for extensions. We constructed a three-electrode electrochemical cell that positions the sample immersed into the electrolyte below the indenter tip prior to the experiment. In addition, the cell allows for sample rotation and electrolyte exchange. The connected Gamry reference 600 potentiostat allows measuring and prescribing currents and voltages. Lubricants can be entered in the cell in a similar way. Supplementary, the G200 allows for the use of multiple indenter tips. Special long indenter tips are used to penetrate the samples immersed into the electrolyte. Conventional diamond tips are used in nanoindentation and nanotribology; here we constructed stainless steel tips to study the steel-on-steel contact and to better understand the five parameters of tribology.

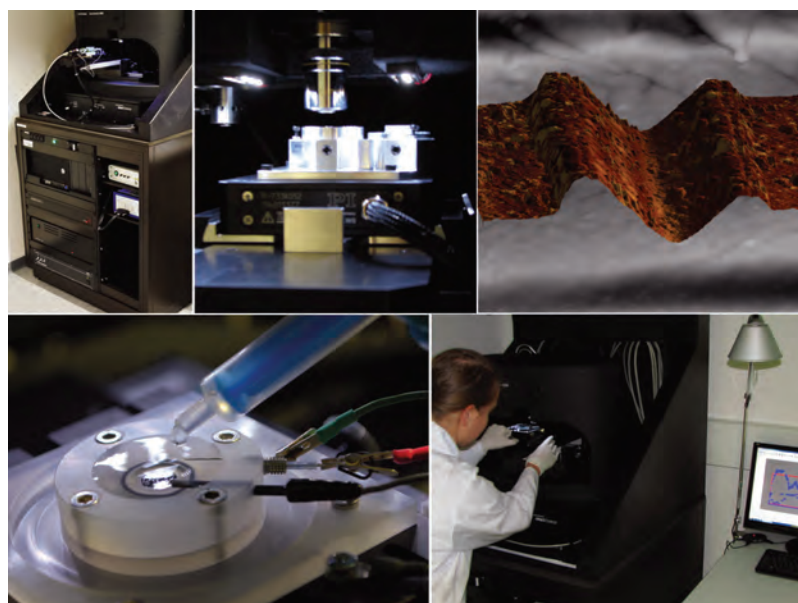


Fig. 1: Snapshots from the Nanoindentation and -tribology Lab: Nanoindenter, nanometer stage, Atom Force Microscopy (AFM) image of a scratch in Cu, electrochemical cell and scratch experiments.

Advanced Transmission Electron Microscopy Facility

C. Liebscher

Department of Structure and Nano-/ Micromechanics of Materials (SN)

Introduction: Transmission electron microscopy (TEM) has a long history and tradition at the MPIE. In the past, the focus was on conventional microscopy, but also more advanced techniques such as scanning TEM (STEM), analytical and *in situ* microscopy became part of research. The Advanced Transmission Electron Microscopy Facility was established in May 2015 with the commission of an aberration-corrected (S)TEM. Correcting for lens aberrations is key to provide researchers with atomic resolution imaging and spectroscopy. The probe-corrected FEI Titan Themis 60-300 combines all state of the art (S)TEM and spectroscopic techniques with an emphasis on scanning transmission electron microscopy. The Advanced Transmission Electron Microscopy Facility will be complemented in mid 2016 by the installation of an image side corrected FEI Titan Themis. The (S)TEM facility enables atomic scale investigation of complex material systems, including interfacial phenomena and precipitate structures. The mission is to resolve the crystallographic structure, chemistry and bonding characteristics of materials developed at MPIE with atomic resolution.

(S)TEM facility: The microscope is equipped with a high-brightness electron source in combination with a Wiener-type monochromator. Reducing the energy spread (<0.2 eV) of the electrons is beneficial for special TEM imaging techniques and mainly for high-energy resolution electron energy loss spectroscopy (EELS). The 3-condenser system allows for nearly parallel illumination in TEM-mode and enables a flexible use of the monochromator in combination with STEM imaging. In STEM, the resolution is mainly limited by lens aberrations of the probe-forming condenser lens. The probe corrector allows for a correction of up to 5th order lens aberrations leading to a STEM point resolution of down to 70 pm. In total, seven STEM-detectors are available for bright-field (BF), annular BF (ABF) and annular dark-field (ADF) imaging. A windowless, four quadrant Silicon-Drift (SDD) EDS (Energy Dispersive Spectroscopy)-detector (FEI Super-X) with a solid angle of >0.7 sr enables atomic resolution

chemical analysis, including light elements like oxygen and carbon. The monochromated electron gun facilitates a flexible adjustment of beam currents up to 2 nA necessary for analytical STEM. Electron energy loss spectroscopy (EELS) can be utilized in combination with TEM or STEM by the post-column energy filter Quantum ERS (Gatan). The integrated electrostatic shutter provides ultra fast acquisition of 1000 spectra/s and dual-channel EELS with simultaneous acquisition of low and core loss spectra. In addition to the CCD (Charge-Coupled Device) of the energy filter, the microscope is equipped with a 16-megapixel CMOS (Complementary Metal Oxide Silicon) camera for high quality, high-speed image acquisition. Precise sample positioning and drift correction is possible through a piezo controlled sample stage. For studying beam sensitive materials, the acceleration voltage can be reduced from 300 to 120 and 60 kV, maintaining sub-nanometer resolution. To overcome the projection problem in TEM, a high-tilt tomography holder with acquisition and reconstruction software is available. The figure illustrates the microscope column and two examples of atomic resolution STEM experiments on SrTiO_3 and a Si grain boundary.

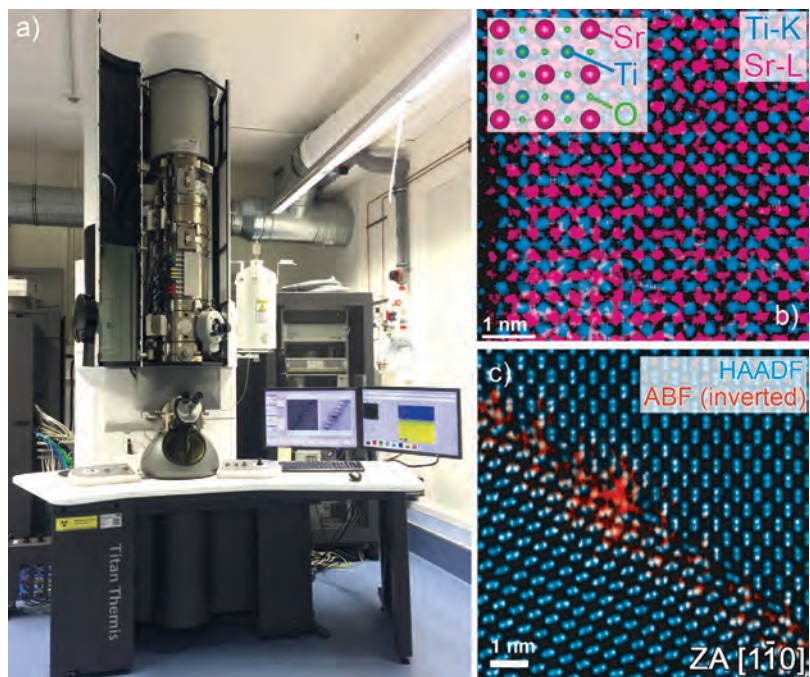


Fig. 1: a) Microscope column of the Cs-probe corrected FEI Titan Themis 60-300 with highlighted features. b) Atomic resolution STEM-EDS elemental mapping of SrTiO_3 . c) Composite image of simultaneously acquired high-angle annular dark-field (HAADF) and annular bright-field (ABF) STEM images of a $\Sigma 3\{112\}$ grain boundary extracted from a intentionally doped multicrystalline Silicon wafer (department of Microstructure Physics and Alloy Design).



The Near Ambient Pressure Microfocus Photoemission Spectroscopy System

D. Vogel

Department of Structure and Nano-/ Micromechanics of Materials (SN)

Chemical reactions at gas/solid and liquid/solid interfaces play a crucial role in many processes, from corrosion to catalysis. A fundamental in-depth understanding of the underlying mechanisms is difficult to obtain due to experimental restrictions for interfacial analysis. The Institute's new near-ambient-pressure X-ray photoelectron spectrometer (NAP-XPS) bridges the pressure gap between UHV (ultra-high vacuum) and ambient pressure, and thus the material gap between simplified model systems investigated under low pressure conditions and complex real systems. Whereas NAP-XPS systems are well established as end stations at synchrotron facilities, our custom-made setup is a prototype of a new commercial laboratory-based system. It enables *in-situ* analysis of surface processes in a pressure range up to 40 mbar at temperatures up to 800°C in an exchangeable high pressure reaction cell with a very small volume.

Thus experiments can be carried out in highly reactive gas atmospheres without contaminating

the UHV chambers or the analyser. XPS and UPS (ultraviolet photoelectron spectroscopy) measurements can be performed using either a monochromatic micro focusing X-ray source, a twin anode (Mg/Al) X-ray source or a small spot UV-source. The system is equipped with devices for sample cleaning, evaporation and characterization such as sputter guns, evaporators or LEED. Additional several bolt-on chambers are equipped to perform pre-treatments or pre-characterizations of the samples, such a high-pressure/high-temperature chamber, electrochemistry cells, or a chamber for performing Kelvin Probe measurements as well as Thermal Desorption Spectroscopy (TDS). The system and its bolt-on chambers are connected to a complex gas installation for cleaning (impurity level < 1ppb) or conditioning of the reaction gases (e.g. defined humidity). The complex installation covers all research fields of the department and offers customized solutions for the related individual projects.

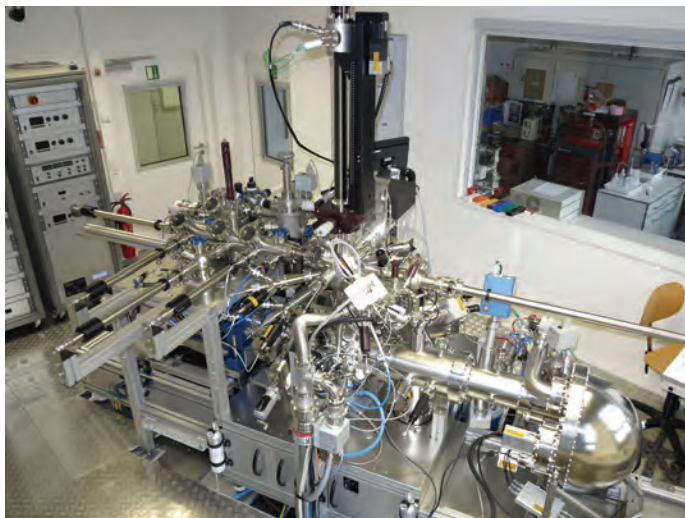


Fig. 1: Photo of the NAP-XPS: The footprint of the system fits ideally into the laboratory, whereas all peripheral devices such as backing pumps, coolers and gas supply are installed in the neighboring technical room.

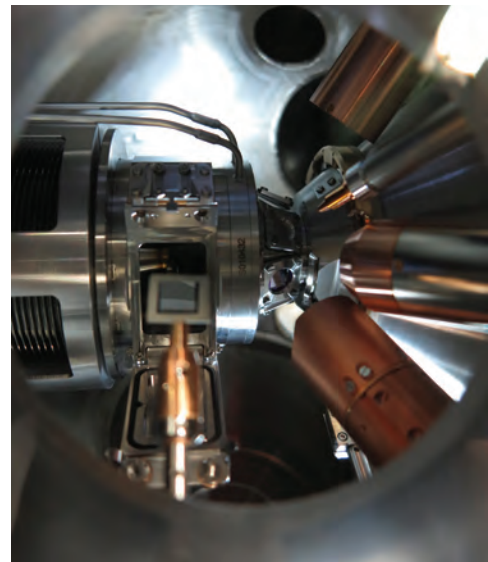


Fig. 2: Photo of the reaction cell inside the UHV chamber: It is docked with the differentially pumped analyzer and opened for sample transfer.

Long-term Oriented Method Development

Development of Electrochemical Water Based *in-situ* TEM and Potential- and Time-Dependent Study of Platinum Alloy Nanoparticle Dealloying

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Platinum is still the best choice for the use as an electrocatalyst in the state of the art proton exchange membrane fuel cell (PEM-FC) [1]. Although its properties and performance have been extensively studied in the last decades, still, its degradation processes in PEM-FC are not completely resolved. Recently, it has been shown that the main degradation mechanisms are Pt dissolution and carbon corrosion [2, 3]. These novel insights were only possible due

to characterization *in-situ* water-based TEM technique, gaining valuable insights into potential- and time-dependent electrochemical processes occurring in materials for energy conversion in their native environment with nanometer resolution. It will enable us in future to obtain the necessary insights and breakthroughs related to for instance corrosion or dealloying of platinum based nanoparticles. We recently utilized a commercial holder for electron microscopy (Protochips Poseidon 510) and presented initial results on platinum alloy nanoparticle dealloying [5]. These experiments helped us to already expose some of the challenges and limits of the studied system, as for instance beam effects and resolution. Next steps include far-reaching *in-situ* experiments in alloy model systems and nanoparticulate catalysts, and we are confident that they will provide new, game-changing insights into fundamental processes on the nanoscale.

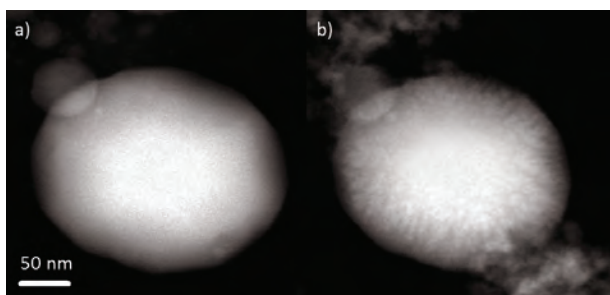


Fig. 1: Electrochemical water based *in-situ* TEM before a) and after b) dealloying treatment of a PtCu₃ nanoparticle in 0.1 M HClO₄. Snapshots out of the *in-situ* video recorded of the dealloying of PtCu₃ nanoparticles in 0.1 M HClO₄ and subsequent copper beam induced redeposition. The big particle is 200 nm in diameter.

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to introduction of new advanced characterization techniques like identical location transmission electron microscopy (IL-TEM [4]) and scanning flow cell coupled to ICP-MS (inductively coupled plasma mass spectrometry) [3] all developed at the MPIE. In order to even further deepen our fundamental understanding especially of structural degradation of catalyst nanoparticles on supports, novel *in-situ* methods will have to be developed and explored.

Bringing together the extensive expertise of the two departments particularly with regards to development of advanced characterization techniques, we plan to develop and utilize advanced electrochemical



Correlative Use of Atom Probe Tomography and Electron Microscopy

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Many fundamental phenomena in materials science such as the segregation to defects, elemental partitioning, nucleation, deformation-driven phase decomposition, or spinodal decomposition need to be understood at the atomic scale, requiring mapping of local chemical composition and crystallography. This requires the development of suited characterization techniques. Atom probe tomography (APT) with its capability of detecting 3D elemental distributions with near atomic spatial resolution and a sensitivity in the range of 10 parts per million, which is independent of the atomic number [1] is currently the best high resolution tool for quantifying local chemical inhomogeneity. However, its spatial resolution is material dependent and often not accurate enough to capture crystallographic aspects such as defect type, grain orientations or phase crystallography. In these cases transmission electron microscopy (TEM) is better suited. But this technique lacks accuracy in quantifying chemical composition, especially in the case of light elements, high dilution and non-planar features. Thus, TEM and APT have complementary strengths and their correlated application to the same specimen provides joint chemical and structural insights to some of the main pending fundamental scientific questions in materials science.

Over the past four years an experimental setup and corresponding know-how has been developed at MPIE rendering correlative TEM/APT experiments feasible and robust [2]. This work led so far to several high impact publications: for the first time nanobeam diffraction (NBD) orientation mapping was used on atom probe tips, thereby enabling the high throughput characterization of grain boundary segregation [3] (Fig. 1) as well as the crystallographic identification of phases [4]. Bright-Field STEM imaging enabled to

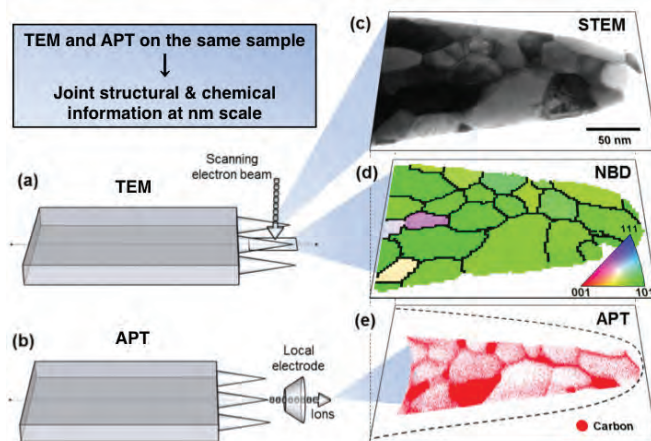


Fig. 1: The correlative use of TEM (a) and APT (b) enables mapping both, structural and chemical information of the same sample at the nanometer scale [3].

prove the existence of linear complexions [5] and to map atomistic details associated with shear-induced mixing [6]. The spectrum of TEM/APT techniques is currently expanded to the correlative use of Dark-Field TEM and APT. Owing to the experimental efforts involved in correlative TEM/APT all scientific cases that do not require the spatial resolution of a TEM should be investigated by scanning electron microscopy (SEM). Therefore, SEM-based approaches to gather crystallographic information on atom probe samples have been developed [7,8]. Future plans involve the use of Electron Channeling Contrast Imaging under controlled geometric conditions (cECCI) [9] on atom probe samples that has the potential to speed up the site-specific sample preparation of crystal defects and their analysis. Intense collaborations with the department “structure and nanomechanics” are planned to combine latest high-resolution TEM instruments with a new atom probe with up to 80% detector efficiency.

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Unified Description of Interfaces between Electron-Conducting Solids and Ion-Conducting Liquids

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Solid-liquid interfaces are at the heart of many modern-day technologies such as water electrolysis and batteries, photo catalytic water splitting, electro catalysis or corrosion. A comprehensive understanding of the full reaction path of the complex electrochemical transformations inherent to any of these processes will aid a systematic development, improvement and optimization of any of them. This requires knowledge of the atomic and electronic structure of the interface and their evolution when environmental conditions change, as well as understanding of the reactivity (e.g. towards oxygen reduction, hydrogen evolution or metal dissolution) as a function of the atomic and electronic structure of a surface/interface. The heterogeneity of the interface and the complexities of the involved solid and liquid materials present a challenge to both experiment and theoretical modelling, requiring the development of experimental techniques which provide microscopic insight and of effective modelling techniques which account for the statistical averaging required to properly describe a liquid, allow reaching the equilibrium state and capture the essential properties of both the solid and the liquid. Hereby, the complementary use of experimental and theoretical approaches is of utmost importance and at the core of ongoing developments within the GO and the CM.

Our endeavour rests on several pillars. Optical experiments investigate the state of water at a solvated electrode/electrolyte interface by vibrational spectroscopy or use adsorption spectroscopy probing electronic transitions to study the defect structure in thin oxide films. Combining experiments with different depth-dependence allows to study electrodes immersed in both thin water films and bulk electrolyte, but also look into intermediate steps by probing solvation using the Surface Force Apparatus (SFA) (few nm), probing solvation in nano-channels (few 10 nm) or probing solvation by angular dependent internal reflection infrared spectroscopy (few 100 nm). Complementary high-resolution force probe and imaging experiments focus onto electric double layer structures. The unique advancement of single molecule desorption studies allow direct measurements of interaction free energies of tethered functional groups with model surfaces [1], of metal interfaces [2] and of arbitrary functionalities. These experiments will be extended to surfaces under varying electrochemical and

environmental (e.g. ΔpH and ΔE) conditions enabling experimental measurement of the interaction free energy of molecules with hydrated electrochemically active interfaces. Several of these experiments provide information accessible by first principles based calculations, which provide the opportunity to benchmark theoretical modelling approaches. Thus, theoretical efforts focus on methodological developments (see p. 161 and [3]) aiming at a realistic description of processes at the solid/liquid interface, but also benchmark individual constituents of the solid/liquid interface or the interface itself, which will provide a firm basis to optimisation of modelling approaches. One example is the effort to acquire accurate ion hydration energies (see p. 75), another, the calculation of spectra for aqueous systems (see p. 29) to probe, e.g., the well characterised phase transition between the pristine and the H covered water/Ge interface. The proper description of electric fields within the constraints given by the periodic boundary conditions and cell-size restrictions of density-functional theory codes will be addressed in the near future.

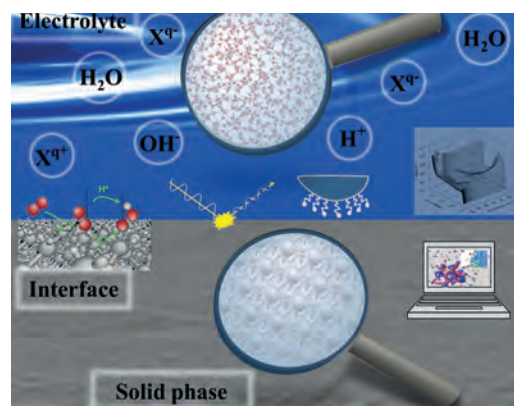


Fig. 1: Schematic representation of the solid/liquid interface. Insets show atomistic details of the constituent condensed phases and interface processes or point at some of the experimental techniques and computational approaches used in our studies.

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Electrochemical Scanning Flow Cell for Combinatorial Reaction Analysis

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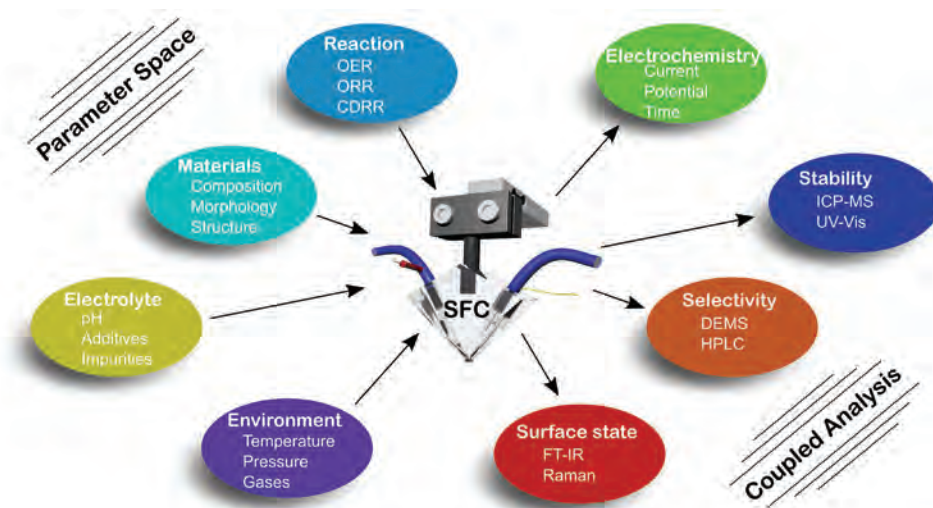


Fig. 1: Concept of the combinatorial reaction analysis by SFC coupled techniques.

In the 21st century the fraction of renewable energy in the overall energy mix will steadily grow. This energy will have to be efficiently distributed, converted, and stored to be utilized to full extent, which can be done with modern electrochemical technologies. In this regard, electrochemistry can be portrayed as a “bridge” between electrical and chemical energy. In reality, to improve the efficiency of energy conversion and thus serve the needs satisfactorily, this bridge requires a support – electrocatalysts. The latter have to be active, stable, abundant, environmentally friendly, and cheap. Understanding the fundamental mechanism and identification of the optimum electrocatalyst for each individual reaction is the main challenge of modern electrochemistry. The most promising tools in this quest are high-throughput experimental approaches, as only such techniques are capable to deal with the complex parameter space that influence electrocatalysis, and can deliver the data required to derive an understanding of the processes involved. The unique electrochemical scanning flow cell (SFC), a workhorse developed in the “Electrocatalysis” group, is a combinatorial approach that can cope with all the requirements and that has been successfully applied for various reaction analyses (Fig. 1) [1].

A typical example that has become increasingly important recently is the oxygen evolution reaction (OER) [2]. Finding optimal material properties and reaction conditions to catalyze the OER is done by fast scanning of material libraries, e.g. thin film gradient samples. The catalyst efficiency is then

estimated directly from electrochemical analysis. Stability information, which is most crucial in the case of the OER, is provided by time- and potential-resolved inductively coupled mass spectrometry (ICP-MS) analysis. As soon as an efficient and stable catalyst is identified, this strategy changes to the investigation of electrolyte properties and operating conditions for the parameter optimization. The obtained information is utilized for the synthesis of high surface area catalyst materials, which are also studied initially with the SFC, and finally are employed in the development of more efficient electrolyzers. To name only a few, other important reactions addressed by the SFC are the oxygen reduction reaction (ORR) for fuel cells, carbon dioxide reduction reaction (CDRR), but also classical corrosion processes [3, 4].

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Scanning Kelvin Probe Techniques

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The recent development at the MPIE to use Kelvin Probe techniques such as Scanning Kelvin Probe and Scanning Kelvin Probe Force Microscopy for spatially resolved high sensitivity measurement of hydrogen has been a major break-through for our work on hydrogen in materials [1]. One big task at the MPIE is therefore to further develop Kelvin probe techniques for hydrogen mapping (see p.39). This success also demonstrated that this contact-free technique for measuring electrode potentials on surfaces and at buried interfaces may hold also further possibilities for exploitation.

One idea is to use the ability to detect hydrogen at very high sensitivity for detecting corrosion sites through the metal, i.e. for instance to measure internal corrosion at the outside of a pipeline or a tank, i.e. through the metal wall. This should allow detecting corrosive attack already before measurable loss of metal. However, for such application a more robust Kelvin Probe device has to be developed that can be used in the field. The development of such a Kelvin probe is a project carried out together with Christian Michelson Research (CMR) institute in Bergen, Norway. From the scientific point of view a number of very interesting issues have to be investigated, such as the detection of hydrogen also through organic coatings and how to deal with trapped charges in coatings.

Another method which is currently being developed is to use the Kelvin probe technique for measuring electrochemical reaction rates at buried interfaces. Up to now this was not possible, although this would be of great importance. For instance, the electrochemical stability of the buried metal-organic coating interface of painted metal is crucially governed by how effectively the oxygen reduction reaction at the interface is inhibited. As this interface is not directly accessible for study by conventional electrochemical techniques, a new non-destructive method has to be developed. For assessing the oxygen reduction rate under buried interfaces we chose to measure the potential resulting from equilibrium between oxygen reduction and hydrogen oxidation. The hydrogen is supplied from the backside of the sample where the hydrogen uptake current is also monitored. One of the crucial points here is to ensure that all the hydrogen produced at the hydrogen entry side is really entering the metal. If so, by measuring the potential at the coated exit side a full I(U)-curve can be thus constructed. Thus oxygen reduction rates at buried interfaces now have become accessible [2].

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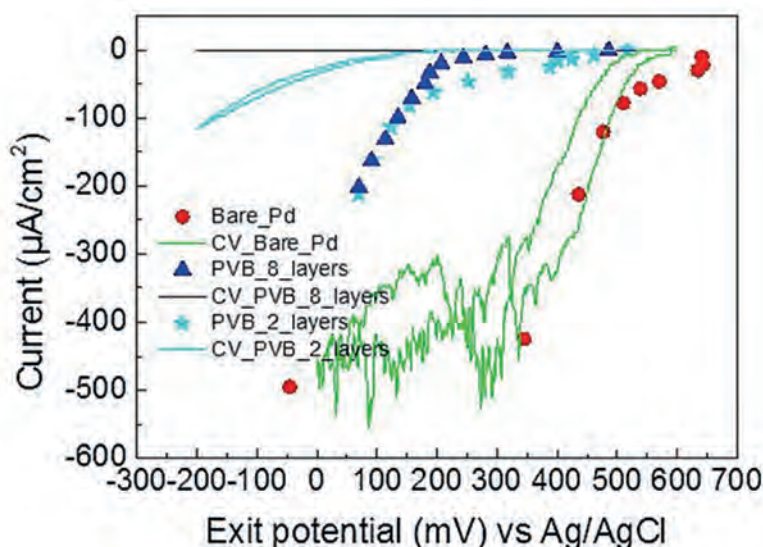


Fig. 1: Cyclic voltammograms (CV) measured on a Pd surface in acidic solution of pH1, bare surface (green solid line), one time PVB coated surface (light blue line), twice coated surface (dark blue line). The symbols show the results obtained by the new methodology. Interesting: the oxygen reduction rate measured with the new technique shows the same rates for the once and twice coated surface. This is to be expected as the interface is the same. The corresponding CVs are different, as these just measure the pin holes and cannot access the buried interface.



Hydrogen Mapping

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From novel high strength steel grades via Ni-based superalloys to modern aluminum alloys, hydrogen related degradation of materials plays an increasingly important role in current materials development. This is not only because they are very susceptible to hydrogen embrittlement, but also because technical application ventures into increasingly aggressive environments, as it is the case e.g. for the oil industry where sour gas environments can lead to very high hydrogen activities. Therefore, the topic is and will remain of crucial importance and is addressed within various publically and industrially funded projects at the MPIE.

energies at different sites by directly measuring the hydrogen release from the investigated material into an ultra-thin palladium detection layer with high local resolution (Fig.1). Hence, a time dependent contrast between the different phases is measured. Up to now the AFM (Atomic Force Microscopy) based Kelvin Probe technique, the so called Scanning Kelvin Probe Force Microscopy (SKPFM), cannot be reliably calibrated and thus only relative hydrogen release rates of different phases and features such as grain boundaries can be obtained.

It is therefore decisive to combine the achieved insights with *ab initio* based simulations of the hydrogen solubility within the different phases as performed by the CM. Using high-throughput concepts, this research considers besides microstructure features also the impact of all alloying elements in the investigated materials.

On the experimental front, SKPFM is currently combined with standard SKP (Scanning Kelvin Probe), which allows the full quantitative measurement of release rates into the palladium layer, in order to obtain highly resolved quantitative information.

In order to obtain direct information about the local concentration of hydrogen, techniques, such as e.g. atom probe tomography or ToF-SIMS are used. As these are surface sensitive techniques most hydrogen that was in the sample during hydrogen charging, will have left the relevant region when the measurement starts, i.e. the measurement will be affected by the effusion

rate from the material, which in turn is measured by the Kelvin Probe techniques. It is the aim of this long-term method development to develop routines for a combination of these experimental and theoretical techniques, in order to achieve a detailed characterization of hydrogen inside complex materials.

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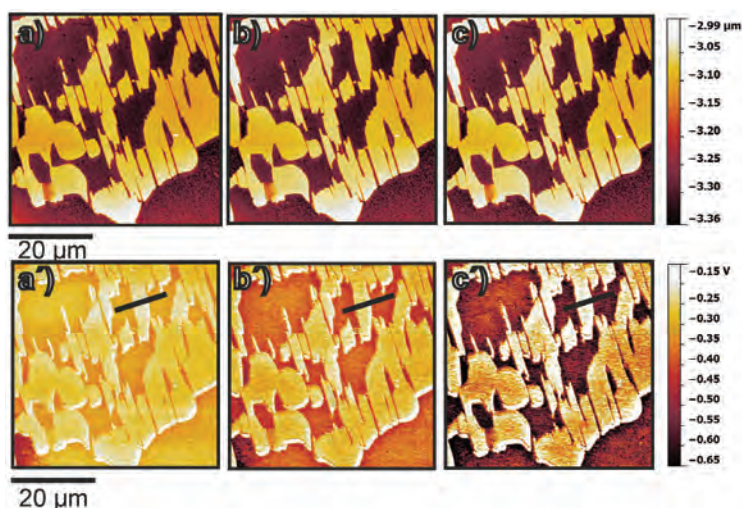


Fig. 1: Results of an SKPFM measurement on a Ni-22wt-% (15at.-%) Nb alloy charged with H and measured 20 h, 50 h and 100 h afterwards. Upper row: topography, revealing that after etching the Ni₃Nb intermetallic phase stands out higher than the Ni matrix. Bottom row: potential difference maps, providing a higher H effusion from the matrix than from the intermetallic phase as the contrast increases with time. Lower potentials correspond to higher H concentration in the Pd layer.

Such projects are needed, since for an in-depth understanding of the degradation mechanisms a detailed characterization and simulation of the materials with respect to their microstructure and of the hydrogen distribution therein are important. For the latter, a number of different experimental techniques are already available at the MPIE, such as the silver decoration technique, microprint technique, tritium autoradiography and time-of-flight secondary ion mass spectroscopy (ToF-SIMS). Additionally, a novel approach based on the Kelvin Probe technique for hydrogen mapping has recently been developed in the GO [1, 2]. Its key strength is to add to a pure H mapping the important information on binding

Grain Boundaries: Interfaces of Outmost Importance in Materials

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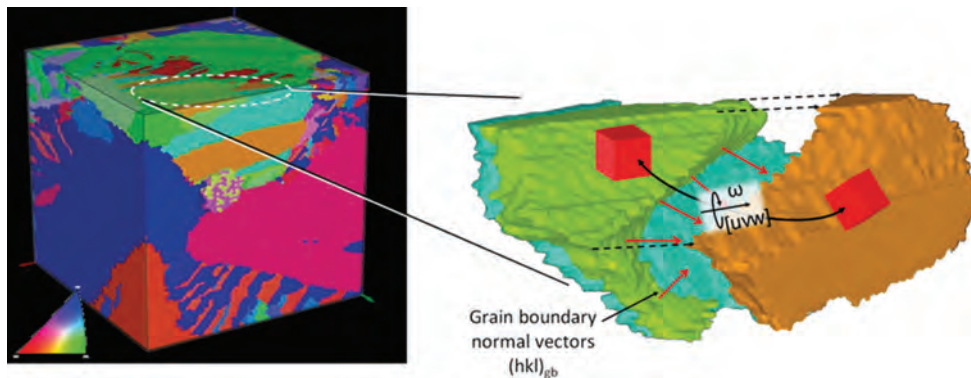


Fig. 1: Three dimensional reconstruction of a polycrystalline microstructure analyzed by 3D-EBSD.

Most structural and functional materials are polycrystalline with grain size and grain boundaries impacting their mechanical behaviour (strength, ductility) [1], corrosion resistance and, e.g., optoelectronic properties. Thus, grain boundaries are a central theme of all departments connecting several research groups at the MPIE.

The bare number of unknowns and variables in one grain-boundary exemplifies the degree of complexity in a nominally simple interface. Crystallographically, the grain-boundary can be described by only five variables: three parameters describing the misorientation in between the two adjacent grains; and two parameters describing the grain-boundary plane. But if it comes to mesoscopic dimensions, it can be noted that most grain-boundaries are not straight, they are curved. A trend which is continuous if zooming into the atomic scale, where atomic positions in the vicinity of the boundary are significantly off their predicted crystalline positions. In terms of pure metals this is the maximum complexity one can possibly encounter. But engineering materials involve another dimension: Their chemical composition across the boundary is far from being homogenous – a fact which opens new routes for tailoring material's properties at all length scales.

During the past years several attempts have been undertaken to get closer to a broad understanding of the role of grain boundaries. Besides advanced microstructural characterization techniques (e.g. TEM (Transmission Electron Microscopy) including High Resolution-TEM, 3D-APT (Atom Probe Tomography), 3D-EBSD (Electron Backscatter Diffraction), μ Laue, *in situ* micromechanics) also simulation techniques bridging across all relevant length scales – from atomistic to macroscopic – had severely been applied to understand grain boundaries.

The MA combines FIB (Focussed Ion Beam)/SEM-EBSD tomography with other tools like cathodoluminescence or local mechanical testing [2] to study the crystallography of boundaries at the mesoscopic level and to obtain, at the same time, information on the properties of these boundaries. APT gives important information on the chemistry of boundaries [3] required to understand, for example, the strength of a material or the formation of new phases at boundaries. Within the SN the work focusses on two questions: (i) can we quantify models for dislocation slip transfer (e.g. [4]) and (ii) what is the impact of dislocation source size distributions as one origin of sample size effects on the dislocation transfer mechanisms. The CM investigates the migration kinetics of grain boundaries in Al using atomistic simulations [5], revealing the fundamental mechanisms involved in the motion of grain boundaries at experimental time/length scales. The cover-picture of this report highlights one of our important findings on the atomistic details of grain boundary migration.

The research activities in the different departments unraveling the important role of grain boundaries are increasingly bundled with new insights by applying complementary experimental techniques and corresponding simulations.

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Electron Channelling Contrast Imaging (ECCI) for Direct Observation of Lattice Defects in Bulk Samples

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Electron channelling contrast imaging (ECCI) is an electron diffraction-based technique for direct observation of individual crystal lattice defects, like dislocations, stacking faults or nano twins, on bulk specimens using the scanning electron microscope (SEM). Although the technique is long time known, it has recently regained interest [1][2], mainly caused by the technological advances in SEM design. In particular, observation of ECC requires an electron beam with very small convergence, small focus point, high beam current and a large but quick detector for backscattered electrons. These contradicting requirements are nowadays satisfied by dedicated SEMs allowing to obtain images of lattice defects with a resolution of better than 10 nm. Though the contrast is less than that in scanning transmission electron microscope (STEM) images, ECCI has the large advantage to give images from bulk samples, looking up to about 100 nm below the surface. An application example for this powerful technique is our recent study on dislocation evolution in a quasi-single crystal superalloy during creep. The observations were done on full cross sections of a test sample in areas of different total stress and strain. As a result it was found that creep dislocations do not only originate from the small-angle interdendritic boundaries,

as it was long time assumed [3], but a significant contribution is made by dislocations nucleating in the interior of the dendrites from individual dislocations being scattered therein [4]. The figure below shows ECCI of dislocations nucleated in the interior of the grain (c) and those nucleated at interdendritic boundaries (d), under a load of approximately 330 MPa. The former ones are indeed much denser. This density difference could also be confirmed by discrete dislocation dynamics simulations carried out for both scenarios.

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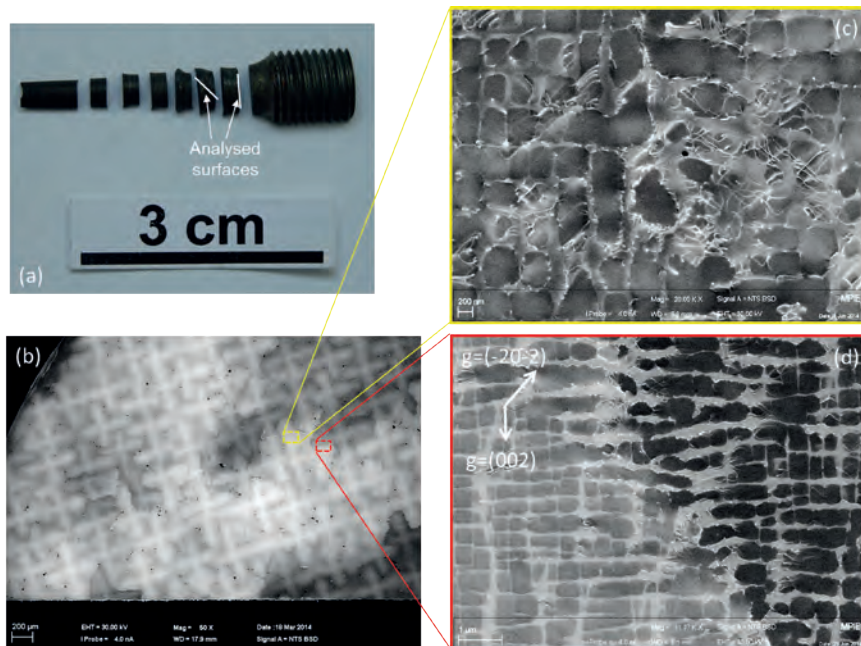


Fig. 1: Dislocation observation in a crept single crystal superalloy test specimen (950° C, 450 MPa), using ECCI. (a) the macroscopic sample, (b) overview on the dendrite structure, (c) dislocations in the dendrite interior, (d) dislocations at a dendrite interface.

New insights into Microstructures by Application of Advanced High-Resolution SEM-Based Diffraction Techniques

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Electron backscatter diffraction (EBSD) is a well-established diffraction technique in scanning electron microscopy. A particular strength of 2D and 3D EBSD-based orientation microscopy is its quantitative nature. In contrast to other microscopy techniques it does not, originally, deliver images but numbers for every scan data point from which images and many other information can be calculated. One example for such a value is the density of geometrically necessary dislocations (GND) that can be calculated as the tensor curl of the 3D orientation field [1]. In contrast, statistically stored dislocations (SSD) are not directly accessible from conventional EBSD although the density of SSDs is as important for evaluation of the total stored dislocation density of a material as that of GNDs. To overcome this deficit a new powerful EBSD analysis method is currently being developed which is Kikuchi band profile analysis based on the newly developed Kikuchi bandlet method [2]. The method functions similar to x-ray peak profile analysis but with the serious complication that Kikuchi lines are created by dynamic rather than kinematic diffraction. Quantification of dislocation densities from these peak profiles is a challenge which we currently

approach by a calibration method using dynamic diffraction-based simulation of EBSD patterns from crystals with defects. A first result of peak profile analysis is displayed in the figure: A peak profile sharpness parameter extracted for various bands of a diffraction pattern undergoes significant changes over a deformed single crystal. This may indicate that different slip systems are active in different areas of the sample, leading to different blurring of different Kikuchi bands in the EBSD patterns. The Kikuchi bandlet method has further attractive applications like improved orientation accuracy, and, potentially, absolute elastic strain determination from individual EBSD patterns [3].

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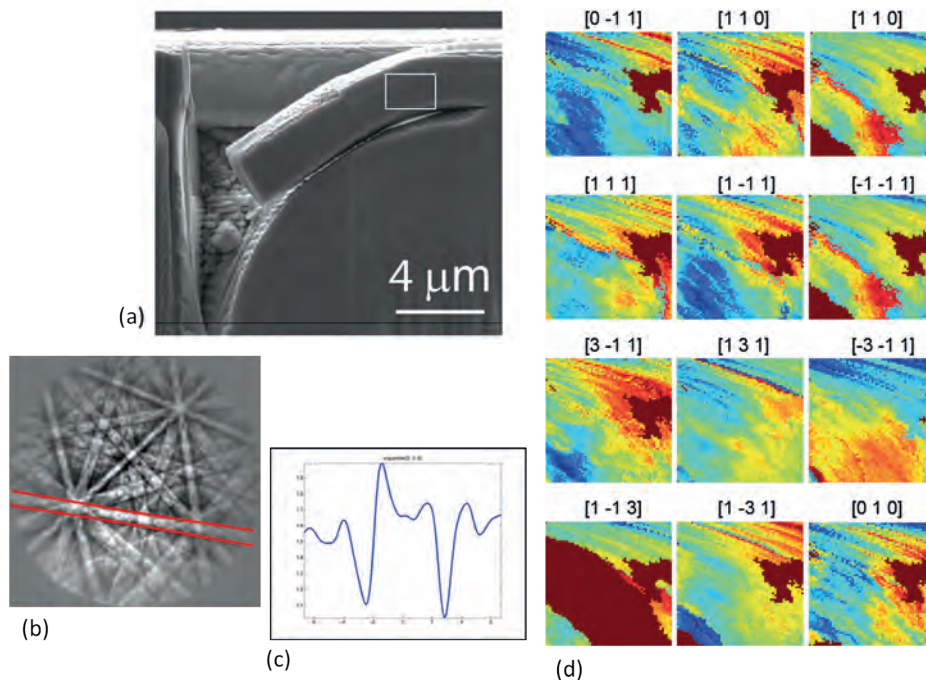


Fig. 1: Plastic strain characterization in a bent microcantilever (a), using the Kikuchi bandlet technique (b) to obtain band profiles of different Kikuchi bands (c). Band profile sharpness maps for different Kikuchi bands are displayed in (d).



Bright Light on Plasticity: *In situ* μ Laue Diffraction

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Interlinking the mechanical response with the underlying and evolving crystallography and microstructure is the key to define material models and to formulate mechanism-based material laws. It requires dedicated *in situ* techniques being able to observe the collective behaviour of a limited number of defects (e.g. dislocations). Within the last years capabilities to deform micron-sized objects in highly brilliant, focused white x-ray light in Laue geometry (*in situ* μ Laue) had been developed and implemented at the French beamline CRG-IF at the European Synchrotron Radiation Facility [1].

μ Laue diffraction is well able to quantify crystallographic phase(s), orientation(s), deviatoric strains and density of geometrically necessary dislocations (GNDs). Our experiments are typically performed with a 500 nm sized white x-ray beam with a spectral range of 5-22 keV. A self-made loading rig was developed at the MPIE and today allows for a displacement-controlled deformation of micron-sized test specimens in compression, tension, bending and fatigue. Typical force and displacement resolution reach micro-Newton and nano-metres. The device operates in full displacement-controlled mode with strain rates from 10^{-4} s^{-1} up to 10^{-1} s^{-1} .

The application of the technique covers several important research topics across the MPIE, like size effects in materials in uniaxial [2] and cyclic loading [3], the dislocation grain-boundary interaction

[4] as well as deformation behaviour of complex microstructures [5,6], investigated together with the department of Microstructure Physics and Alloy Design. Thus, μ Laue diffraction is one of the corner stones of the SN and micromechanics group.

In future, the main focus is to put on the improvement of data interpretation (“3D-EBSD informed Laue analysis”) as well as implementation of non-ambient temperatures and media in the straining device.

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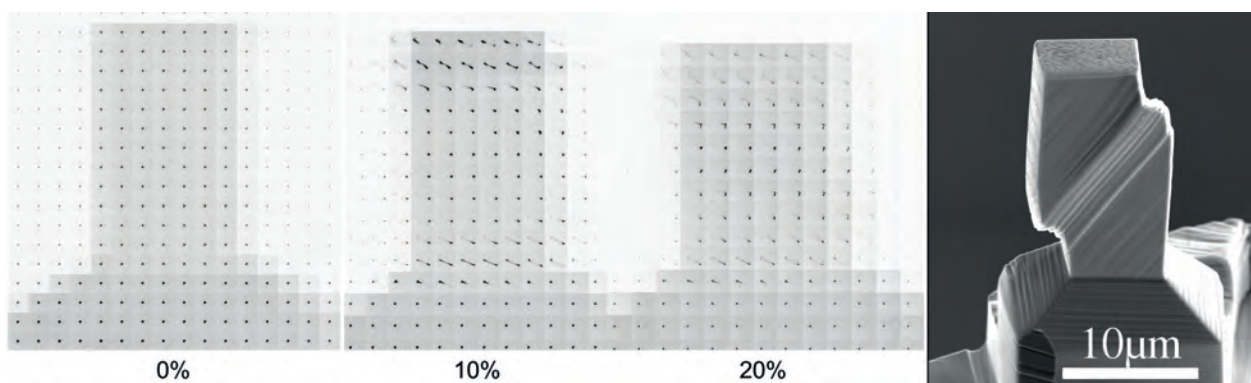


Fig. 1: μ Laue composite images of a copper micro pillar. The x-ray beam is scanned across the entire $7 \times 7 \times 21 \mu\text{m}^3$ sized samples and the representative (111) reflection at various locations is plotted. At 0% engineering strain (left) the spots are all circular, indicating the lack of stored GNDs. During stepwise straining to 10% and 20% GNDs are stored in the top and bottom part to accommodate for experimental constraints. The scanning electron microscope (SEM) image to the right shows that the deformation is confined to only few slip steps, documenting that the two methods are complementary: μ Laue probes stored dislocations, SEM slip step analysis not.

Innovative Material Synthesis

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Within the group Combinatorial Metallurgy and Processing about 500 metallic materials are synthesised every year for both internal projects – with all departments – as well as external cooperations with scientific and industrial partners. This represents the basis to investigate micro- and nano-structural phenomena, validate simulations and study complex transient phenomena such as non-equilibrium phase transformations and interfacial reactions. Accordingly a wide range of experiments are performed, from highly controlled syntheses of single- or oligo-crystalline materials, to binary model alloys, as well as modified industrial materials. The corresponding solidification kinetics can be controlled from quasi-equilibrium conditions up to extremely rapid cooling required e.g. for amorphous metallic glasses.

The spectrum of the advanced structural materials ranges from novel lightweight titanium- or magnesium alloys, to intermetallic materials and high entropy alloys, which is made possible by the high level of qualification and experience of the technical personnel. The main expertise, however, lies on the synthesis and processing of innovative iron based materials, such as lightweight-construction steels [1]. Currently, the main focus lies on *in-situ* metal-matrix-composite steels with an improved stiffness / density ratio [2], also termed high modulus steels (HMS). We recently demonstrated how particles such as titanium

diboride can be refined down to the nanometric range with liquid metallurgy synthesis production techniques, thus overcoming the inherent conflict between physical and mechanical properties (Fig. 1).

The design of this multitude of novel materials goes hand in hand with the development of innovative synthesis and processing routes with differing scopes [3], all embedded into developments stretching over a long term and in strong interaction with other groups and departments. Innovative methods for the accelerated synthesis, processing and testing of bulk metallic structural materials are developed and deployed, thus reducing the time between an alloy design idea and the final evaluation of the materials' mechanical and microstructural properties from several weeks or even months down to hours [4].

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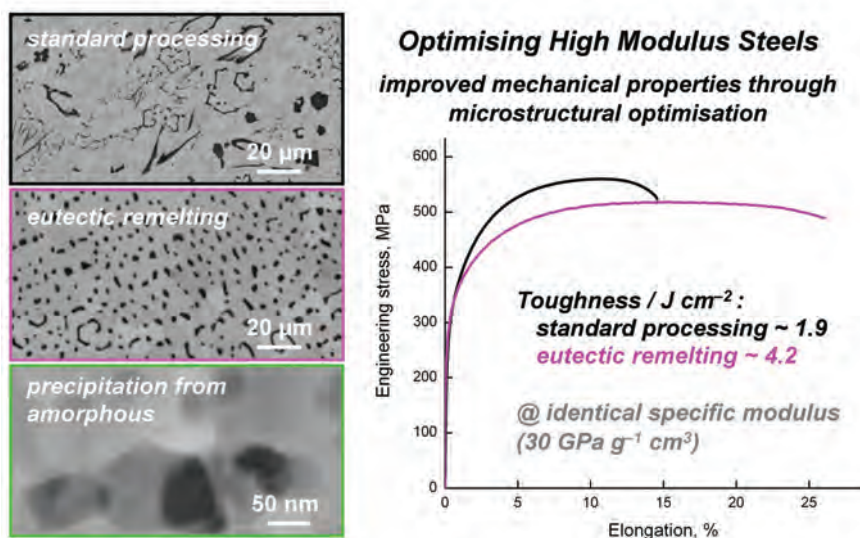


Fig. 1: Innovative material synthesis, here exemplified on the optimisation of stiff and lightweight high modulus steels: improved mechanical performance (right) through refinement of shape, size and dispersion of TiB₂ particles in Fe matrices (left, dark particles) by controlled solidification kinetics.



The Düsseldorf Advanced Material Simulation Kit: DAMASK

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Crystal plasticity modelling has matured into a cornerstone of computational materials science [1]. Developing this field from its original mean-field homogenization approach in conjunction with viscoplastic constitutive hardening rules into an advanced multiphysics continuum field solution strategy requires a long term initiative. Roter's "Theory and Simulation" group is working in this field since 2000. Starting originally from Kalidindi's numerical crystal plasticity integration scheme [2] user subroutines for the commercial FEM (Finite-Element-Method) packages Abaqus and MSC.Marc were developed. The scientific focus of the group was, however, the development of advanced constitutive models based on dislocation densities as internal state variables, which are capable of providing microstructure based predictions [3]. As simulations of different material classes and on different length scales necessitate the use of constitutive descriptions of varying degree of sophistication, it soon turned out that the numerical implementation was not flexible enough to incorporate different constitutive models. Therefore, within the project "Computational Modelling of Polycrystals" (CMCⁿ, the first joined MPG-FhG project ever, established in

2006) the development of a flexible framework, for crystal plasticity simulations on all scales from the single crystal up to the engineering component, was initiated. The new code is strictly modularized to allow easy incorporation of additional models on all length scales. In this way, two different homogenization schemes and seven different constitutive models for plasticity, including a non-local one treating dislocation fluxes [4, 5], have been incorporated into the code package. The ability to use different constitutive models within a single simulation is a unique feature of the code. In 2010, in collaboration with Prof. R. Lebensohn (Los Alamos National Laboratory, Humboldt awardee at MPIE at that time), a spectral method based boundary value problem solver was added to complement the commercial FEM solvers. In 2011, it was decided to release the code, by now called DAMASK, as free software to the public domain. For that purpose, a website (damask.mpie.de) was launched in September 2011. The science community very well received the idea of a free crystal plasticity code. At least 15 groups, including universities like the University of California, research facilities like the Los Alamos National Laboratory as well as companies like Tata Steel (IJmuiden), today use DAMASK worldwide (as it can be freely downloaded we do not know the real number). These groups contribute to the further code development as well as adding new features to DAMASK including new/modified constitutive models [6]. Currently, DAMASK is extended towards a multi-field solver. This will allow treating coupled problems, e.g. thermo-mechanical-chemical, in a fully consistent way.

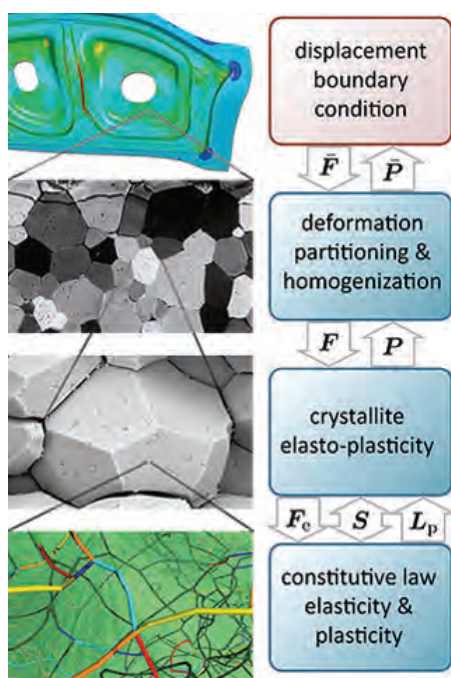


Fig. 1: Hierarchy of scales as treated in DAMASK.

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Ab initio Thermodynamics for Materials Design

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A central aim of the CM is the development and application of techniques that allow for an efficient and accurate computation of thermodynamic properties at finite temperatures from *ab initio*. A severe challenge in such computations is that free energies of the various phases are almost degenerate, often requiring to resolve energetic changes below 1 meV/atom. The theoretical formalisms therefore need to accurately capture all relevant entropic contributions due to electronic, vibrational or magnetic excitations, as well as their coupling such as phonon-phonon, magnon-phonon interactions or spin-quantization.

Over the past ten years the CM has combined accurate first principles calculations with advanced statistical sampling methods allowing now to address these issues and to determine free energies and derived thermodynamic quantities that often rival available experimental data. Our efforts recently allowed us to develop and implement a wide range of optimized methods for various tasks such as, e.g.:

- the random-phase-approximation upsampled thermodynamic integration using Langevin dynamics (RPA UP-TILD) to include electronic correlation effects in finite temperature calculations [1],
- the local anharmonic (LA) approximation to highly efficiently capture anharmonicity of solids with a few $T = 0$ K calculations only [2],
- the two-stage upsampled thermodynamic integration using Langevin dynamics (TU-TILD) method to accelerate anharmonic simulations of arbitrary structures and phases [3],

- the local Grüneisen theory (LGT) to accurately capture the observed non-Arrhenius behaviour for point defects [4],
- the extended spin space averaging (SSA) method to describe magnetic disorder [5].

Besides providing an efficient and accurate sampling of the phase space, these approaches give also a direct insight into the fundamental physical mechanisms responsible for the finite temperature energetics. A few selected applications are exemplified in the figures.

Long term developments in this field will be focused on the extension of these methods to alloys, extended defects, and liquids, all of which are decisive for the understanding and design of structural and functional materials.

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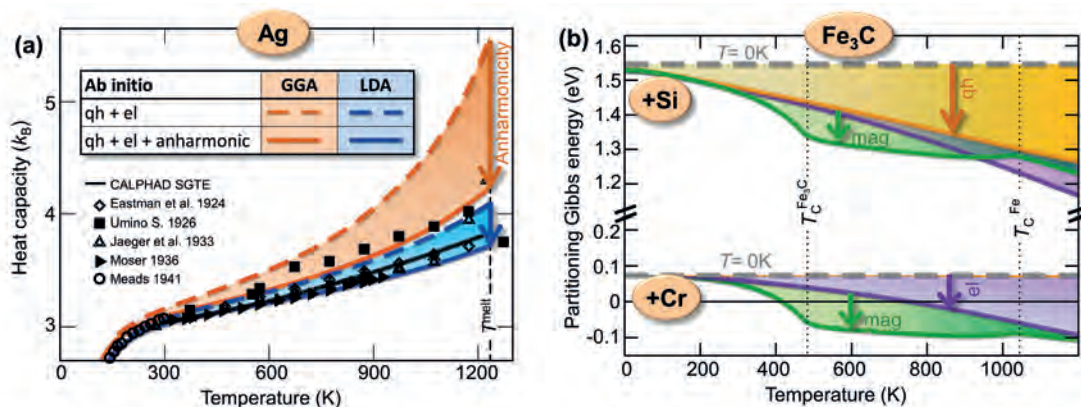


Fig. 1: Recent examples for *ab initio* thermodynamics: (a) Impact of anharmonic phonon-phonon interactions (shaded region) on the heat capacity of Ag [1]. (b) Partitioning Gibbs energies between ferritic iron and cementite for Si and Cr. The influence of vibrations (qh), electrons (el), and magnetic (mag) excitations is shown and the Curie temperatures of cementite and ferrite are marked with the dotted lines [6].



Large-scaled and Networking Projects

The International Max Planck Research School SurMat – Interface Controlled Materials for Energy Conversion (IMPRS-SurMat)

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

E. Gattermann, A. Erbe

The IMPRS-SurMat is a structured, three-year doctoral programme coordinated at the Max-Planck-Institut für Eisenforschung since 2004. During the second running period (till December 2015) the focus was broadly on materials interfaces. Research groups from the Ruhr-Universität Bochum, the Interdisciplinary Centre for Advanced Materials, the Max-Planck-Institut für Kohlenforschung and the Max-Planck-Institut für Eisenforschung brought their various competences together, and they lead interdisciplinary projects to outstanding results. The close link between experimental and computational science enhanced this effect.

In April 2014, the IMPRS-SurMat was very successfully evaluated. The external reviewers were impressed by the excellent scientific output by the IMPRS-SurMat students and by the future concept. Further, all partners assured a substantial own financial contribution. The new concept, briefly outlined below, also convinced the state of North Rhine-Westphalia to contribute financially to the new school. All reviewers recommended therefore the continuation of the school. The responsible

committee of the Max Planck Society followed this recommendation and the IMPRS-SurMat got extended for its third period that will last from 2016 till 2021. Consequently, in January 2016, the third running period of the International Max Planck Research School SurMat begins. This prolongation will bring some major changes.

During the last years, new key aspects came into the focus of all partners. Many groups work now on topics important for the development and understanding of materials for energy conversion. Two further institutes of the region are dealing with these aspects: the Max-Planck-Institut for Chemical Energy Conversion and the University Duisburg-Essen. Groups from both institutions could be won to join the IMPRS-SurMat and will officially join the programme with the beginning of the new period in January 2016. With this step, the main players of the Rhine-Ruhr region in the field of materials interfaces are coming together in the IMPRS-SurMat.

The scientific topic “Interface Controlled Materials for Energy Conversion” shall combine the strengths of the partners and focus on four topics:

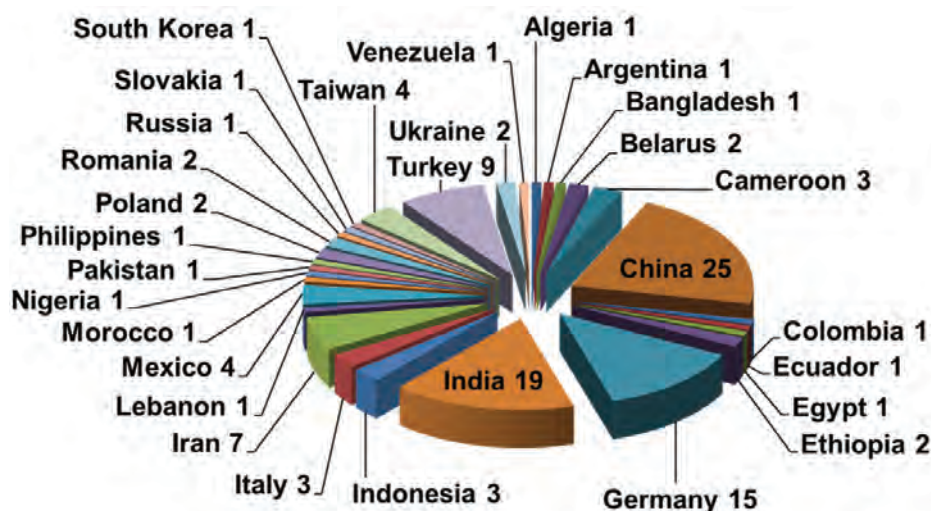


Fig. 1: Origin of IMPRS-SurMat students by countries.

- Microstructure and physics of defects
- Hydrogen and oxygen technology
- Materials for future energy systems
- Degradation mechanisms and life extension of materials

In April 2015, a kick-off workshop for the new period took place at the MPIE. The professors and departmental directors of the current and the new partner institutes presented their research fields and how they plan to contribute to IMPRS-SurMat projects. The IMPRS-SurMat students presented the current research work in a poster session. This fruitful workshop lead to the start of several new projects already in 2015.

For the doctoral students, the programme continues to offer a well-balanced curriculum and ensures the best available supervision. The programme is conducted entirely in English and the doctoral degree (Dr. rer. nat or Dr. Ing.) is conferred by one of the partner universities. The school offers funding for three years, but is also open to students funded from outside as long as all required criteria are met.

The core curriculum remains to be delivered over years one and two. It is structured as a series of four two-week classes each term (winter/summer) that explore various aspects of modern materials and interface science. In addition soft skill trainings, such as presentation skills, scientific writing, project management, and career development events – the latter in the form of career talks given by industry contributors - are taking place regularly.

Each project is supervised by at least two scientists from different departments and institutes. Thus, the interdisciplinarity is guaranteed. Once per year, the doctoral students send a short report about the progress of their work to the programme coordinator. Subsequently, Thesis Advisory Committee (TAC) meetings take place to discuss the results and plan the next working steps. Previously, TAC meetings took place only after the second year.

From 2004 on, 118 students from 28 different countries joined the IMPRS-SurMat. Most of them came from Asia (China, India, Iran), but there are also students from Europe, Africa, Middle and South America. To get admitted the students have to apply online in one of the two application rounds taking place per year. The application process has continuously evolved into an efficient selection procedure. All shortlisted applicants are invited for personal interviews. After a descent of admissions in 2014 because of the evaluation and planning of the new period, already 15 new students were admitted in 2015 to start with the new concept and to integrate the new partners.

Since 2004, 65 students have successfully finished their doctorate within the IMPRS-SurMat (23 students from 2013 till 2015), the duration of the doctoral work is in the most cases between 3 and 4 years. The scientific output is genuinely high. Besides more than 170 poster and oral presentations on conferences, 287 journal articles have been published.

With the new partners and the new concepts, we are looking forward to a successful third running period of the IMPRS-SurMat.

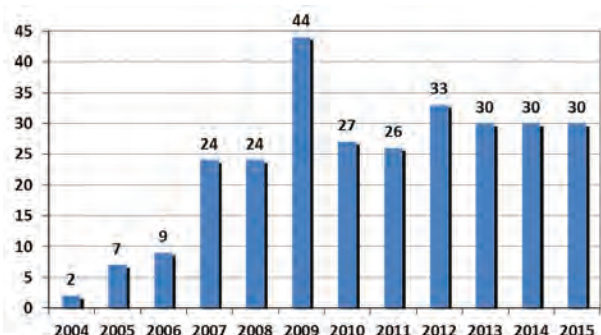


Fig. 2: Number of publications by IMPRS-SurMat students in peer reviewed journals.



Fig. 3: IMPRS-SurMat community at the annual retreat in 2014.



Gas Turbines of Tomorrow: From Atom to Turbine Blade – Scientific Foundations for a New Generation of Single-Crystalline Superalloys

SFB/TR 103

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Ni-base superalloys are the key engineering materials for high-temperature applications, such as aircraft engines or land-based turbines for electrical power generation. The collaborative research centre SFB/Transregio 103, devoted to comprehensive understanding of superalloy structure and properties, was established in 2012 at the Ruhr-Universität Bochum and the Friedrich Alexander University of Erlangen-Nürnberg. The MPIE participates at the SFB/Transregio 103, performing 3D characterization of alloy chemistry by means of atom probe tomography (APT). This method provides essential information on elemental partitioning and segregation within the microstructure

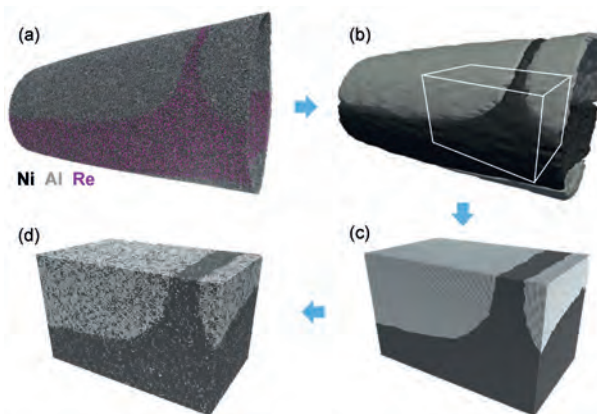


Fig. 1: Generation of atomistic simulation samples from APT data. (a) Reconstructed APT specimen (only Ni, Al and Re shown). (b)-(d) Use of reconstructed iso-density surfaces of Al (gray) and Re (black) for developing a non-stoichiometric APT-informed atomistic sample with realistic atomic configurations.

of Ni-base and novel class of Co-base alloys, on elemental redistribution under creep deformation and formation of deleterious topologically close-packed (TCP) phases.

Experimental APT data for an industrial CMSX-4 alloy were used for generating a realistic sample for atomistic simulations (Fig. 1) in order to study the interaction between γ -channel dislocations and γ' precipitates [2]. It was shown that the precipitate morphology, in particular its local curvature, and the local chemical composition significantly alter both the misfit dislocation network at the precipitate interface and the core structure of the misfit dislocations. APT-informed simulated tensile tests reveal the

atomic scale details of many experimentally observed dislocation-precipitate interaction mechanisms, which cannot be reproduced by idealized simulation setups with planar interfaces.

Precipitation of TCP phases, which have a detrimental effect on creep lifetime of alloys, was another important topic. Ru is known to significantly slow down TCP phase formation, but the exact mechanisms of the Ru effect on elemental partitioning and TCP phase precipitation have been under debate for a long time. For a series of complex Ni-base superalloys, Ru was shown to partition equally to γ and σ (TCP) phases and significantly reduce TCP precipitation. However, Ru did not lead to notable changes in partitioning of other elements or to their segregation at phase boundaries [3, 4]. The earlier proposed hypothesis of ‘reverse partitioning’ (redistribution of Re content from γ to γ' caused by Ru) was disproved and an alternative mechanism proposed, which involves an increase in lattice misfit between the σ -phase and the parent γ -phase in the presence of Ru and resulting in an increase in nucleation barrier.

Besides Ni-base alloys, a new class of γ' -strengthened Co-base alloys was investigated with a focus on the influence of alloying elements on the microstructure and mechanical properties. The elemental partitioning of Al, W, Ti, Ta, Ni and Cr and their mutual influence was studied [5, 6]. The positive effect of Ti and Ta on stabilization and mechanical properties of the γ' phase due to their strong partitioning to γ' was established. On the other hand, Cr had a positive influence on γ' formation but a negative effect on the creep resistance, which was associated with its influence on the stacking fault energy of the γ' phase.

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SMARTMET: Utilizing Phase Instability to Design Alloys with Enhanced Mechanical Stability

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In 2011, Prof. D. Raabe and Prof. J. Neugebauer were awarded an ‘ERC Advanced Co-Investigator Grant’, the most prestigious and competitive scientific funding in Europe. The project proposal: “SMARTMET: Adaptive nanostructures in next generation metallic materials” was embedded into the Adaptive Structural Materials (ASM) group lead by Dr. C.C. Tasan (experiments) and Dr. B. Grabowski (theory).

The main aim of SMARTMET is to address the inverse strength-ductility problem, which sets an apparent limit to the mechanical optimization of advanced engineering alloys. The basic solution approach proposed therein is the joint use of experiments and atomic-scale theoretical tools to develop new alloy design strategies based on utilizing phase instability.

A main activity combining experimental and theoretical efforts focused on designing titanium based alloys at the verge of their stability. Such ‘metastable’ Ti-alloys show tremendous potential for biomedical, functional and structural applications, but exploiting the potential has so far been hindered by the intrinsic microstructural complexity revealed by these materials, as well as absence of proper guidelines in determining ideal compositions.

The theory and experimental teams have jointly selected gum metal as a starting point, an alloy with excellent properties but strongly debated micro-mechanisms, for investigating the potential for instability-based property improvement.

The experimental team has confirmed, through an extensive multi-scale characterization effort, that this alloy demonstrates multiple, previously unknown, phase transformation phenomena: i.e. a reversible nanotwinning-assisted backpack transformation from β phase to α'' and to ω (Fig. 1a).

To understand the physics of these transformations, we referred to *ab initio* simulations. The calculations

lead to the fundamental understanding on the electronic scale of the phase stabilities in Ti and its alloys. Based on this knowledge and on the in parallel developed finite temperature methodology we were able to compute phase diagrams in various Ti alloys (Fig. 1b). In particular, we show that at certain compositions a triple point occurs at which the phase fields of the β - ω - α'' phases are connected. This feature has important technological consequences, because it predicts compositions ranges for designing special multiphase Ti alloys.

This work is only one example of the research activities in the ASM group. There are various phase stability studies on other alloy systems, e.g. high entropy alloys, advanced multi-phase steels, Ti-alloys, etc., which demonstrate the power and the versatility of the “SMARTMET approach” of utilizing instability.

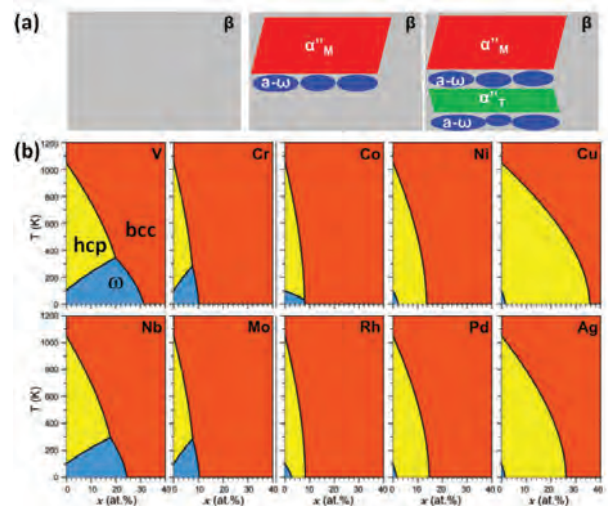


Fig. 1: (a) Schematic representation of the nanotwinning-assisted backpack transformation phenomena; (b) Various Ti-X phase diagrams determined by *ab initio* simulations.



International Network Mechanics of Nano-Objects - MECANO

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MECANO is a multinational and interdisciplinary network, which brings together physicist, chemists, materials scientists and mechanical engineers to advance the understanding of mechanical properties of materials at small length scales. The network, which was established in 2012 by Prof. Olivier Thomas (Marseilles), is funded by the French research organization CNRS for 4 years and comprises researchers from Austria, France, Germany, Ireland, Italy, Switzerland, and UK and includes partners from universities, research institutes, large-scale research facilities, and industry. MPIE is one of the 11 European partners and actively involved in operating MECANO. G. Dehm is a member of the Scientific Committee, which meets twice a year to decide on topics of forthcoming meetings, workshops and schools. The scientific questions addressed within MECANO stretch from synthesis of nanomaterials, manipulation of their properties, to advanced experimental testing and characterization methods for quantitative determination of stress, strain, and defects down to the atomic level, questioning – as always in nanoscience – how well current theories used at the macro- and mesoscale (continuum

mechanics, plasticity, elasticity theory, ...) can be applied to smaller and smaller length scales. The portfolio of topics also encompasses reliability of devices and enhancement of their performance by strain engineering. The network is structured into four themes:

- Mechanics in small dimensions: elasticity, plasticity and fracture
- Experimental methods: local fields mapping and mechanical testing
- Modelling and simulation: from Ångstroms to microns
- Coupling between growth, stress and composition in nano-objects

These four topics span the core research interests of the network members and are strongly interlinked.

The complexity of the topics demands an interdisciplinary approach bringing together experimentalists and theoreticians. The strength of MECANO is the networking across discipline boundaries involving scientists across all levels of their career from graduate students to senior scientists. The MECANO network promotes scientific discussions and fosters collaborations by organizing (i) General Meetings open to all topics, (ii) dedicated Workshops on specific topics, and (iii) Schools for graduate students to review basics and put recent research developments into perspectives. MPIE organized the 2nd General Meeting from 18-19 July, 2013 with ca. 70 participants (Fig. 1). Two Schools were organized on Small Scale Plasticity (Cargese, 14-19 October 2013) and Solid Mechanics for Nanoscientists (Autrans, 17-25 March 2014) and attended by PhD students from MPIE. The international MECANO network has been very fruitful and a new funding application for the period 2016-2019 is currently compiled by the Scientific Committee.



Fig. 1: Participants of the 2nd General Meeting held at the MPIE in Düsseldorf from 18-19 July, 2013 with ca. 70 participants including 24 PhD students and 13 postdocs.



Steel - *ab initio* - Quantum Mechanics Guided Design of New Fe-based Materials: A Joint Initiative between MPIE and RWTH

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In 2007 the MPIE in Düsseldorf and RWTH Aachen University jointly initiated a collaborative research centre (Sonderforschungsbereich, SFB 761) on quantum-mechanics guided design of new Fe-based materials. The initiative is funded by the German Research Foundation (Deutsche Forschungsgemeinschaft DFG). 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). The results of the second four-year phase of the SFB 761, which ended in June 2015, were successfully presented during the evaluation of the SFB in early 2015. Based on this evaluation and the written report, a third four-year phase with seven projects executed at MPIE was granted by the DFG.

The key idea of the SFB 761 is to develop a new set of methods for materials and process design based on *ab initio* calculations in conjunction with advanced characterization and metallurgical alloy development tools. The first phase of the project focussed on the ternary Fe-Mn-C system, forming the basis of high manganese steels. During the second phase the alloying spectrum was extended to Fe-Mn-Al-C. A key quantity in the investigations was the stacking fault energy, which serves as a central link between local chemistry and engineering applications. It was used as an input in physically based strain hardening models that are fully coupled to finite element simulations. Such a multiscale approach was for example applied to investigate the crashworthiness of TWIP steel and resulted in an excellent agreement with the crash performance experimentally observed for a design component. Breakthroughs have also been achieved in the processing of TWIP steels by strip casting and in the combination of high-resolution characterization techniques such as transmission electron microscopy (TEM) and atom probe tomography (APT) to investigate for example the C segregation behaviour.

Now, in the third phase, the material spectrum is again extended towards steels with either medium Mn or higher Al content. This opens another spectrum of physical questions, where in particular

phase boundaries (α/γ and γ/κ) become important. Other focus topics of the third period are strain hardening engineering and hydrogen management in these steels.

The new methods and insights developed within the SFB 761 have an impact beyond the project consortium. Within a first transfer project with an industrial partner, the tools have been applied to Fe-Cr-Mn-N-(C) steels. In the third period, a couple of further transfer projects are established. One of them, which is run by MPIE, evaluates the sensitivity of high-Mn steels that contain Cr carbides to hydrogen embrittlement. Interfaces are also of central importance in this project.

Inspired by the success of the first two periods, the SFB 761 will continue in the manufacturing and characterization of Fe-Mn-Al-C-steels of different compositions. *Ab initio* methods are not only used for the prediction of key thermodynamic parameters, but turned out to become increasingly important for understanding the ongoing mechanisms (relevant, e.g., for TWIP, TRIP, weight reduction, shear band formation and kappa-carbide formation). The aim is the quantification of the effects of chemical composition, strain rate and temperature on the occurrence and interaction of different strengthening mechanisms. The long-term perspective lies in the development of predictive and quantitative multiscale models of materials and processes that are based on *ab initio* simulations and the establishment of a new class of structural steels based on the Fe-Mn-C system.

More details on SFB761 are available on

<http://www.stahl-abinitio.de>



Fig. 1: The SFB 761 team starting into the 3rd period in July 2015.



ICAMS - Interdisciplinary Centre for Advanced Materials

J. Neugebauer ¹, T. Hickel ¹, R. Spatschek ^{1,2}, D. Raabe ³

¹ Department of Computational Materials Design (CM)

² now at: Forschungszentrum Jülich

³ Department of Microstructure Physics and Alloy Design (MA)



The Interdisciplinary Centre for Advanced Materials Simulation (ICAMS), which has been initiated in 2005 by the MPIE and has been founded in 2008, has become one of the leading institutions for scale bridging materials modelling. It includes the departments for Atomistic Modelling and Simulation (R. Drautz), Scale Bridging Thermodynamic and Kinetic Simulation (I. Steinbach), and Micromechanical and Macroscopic Modelling (A. Hartmaier). After the

which is financially supported by industry, works on the efficient implementation and parallelization of various codes developed at ICAMS. The ASG “Diffusion and Microstructure Analysis” (G. Wilde) brings in complementary experimental expertise on topics such as grain boundary diffusion, internal interfaces, as well as microstructure evolution in deformed materials.



Fig. 1: Vivid discussion at the meeting of the ICAMS advisory board (headed by Dierk Raabe), with members also from the ASG Modelling from the MPIE.

The link between ICAMS and the MPIE is further deepened within two large-scale projects financed by the German Research Foundation (DFG) that have been attracted in the past years: Within the Collaborative Research Centre (SFB/Transregio 103) “From Atoms to Turbine” joint research on the characterization of superalloys is performed. The MPIE is further participating in the Priority Programme (SPP1713) “Chemomechanics” that is coordinated by I. Steinbach. Its associated projects investigate the interplay of mechanical and chemical forces in metals and polymers. Various other projects, e.g. within the Max-Planck Research School SURMAT, have led to an increasing amount of PhD theses that are jointly supervised by members of both institutions. Also, lectures within the ICAMS Master of Science programme “Materials Science and Simulation” (MSS) are partially held by members of the MPIE.

initial startup period of five years with substantial financial support from an industrial consortium, the state of North Rhine-Westphalia and the European Union, the institute has now become a regular unit within the Ruhr-Universität Bochum. Due to its excellent scientific performance, documented for example by its international outreach, a large number of publications, as well as a significant amount of attracted third-party funding, there is no doubt that ICAMS can successfully continue its work in the future. The MPIE has served as a strong scientific partner in this development.

Due to the scientific exchange in jointly organized workshops (e.g. Ringberg Unary Workshop 2013, ADIS workshop 2014) and symposia at international conferences, common retreats, and the ICAMS Advanced Discussions, it is ensured that the close collaboration between the two institutes will also flourish in the future.

Structurally, the MPIE is embedded into ICAMS via the Advanced Study Group (ASG) “Modelling”, which supports the multiscale concept in particular at the most fundamental scale, which is dominated by electronic interactions and individual atomic processes. The success of the ASG concept has recently led to the integration of two further ASGs: Since 2012 the group “High Performance Computing in Materials Science” (G. Sudmann),

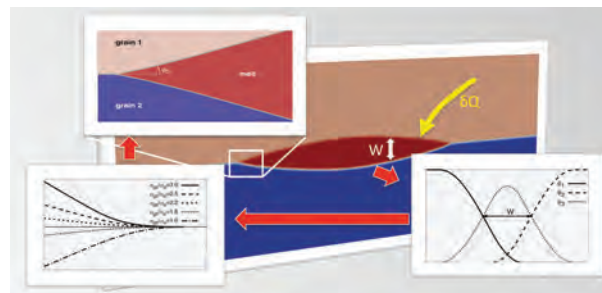
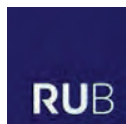


Fig. 2: Modelling of melting along grain boundaries as a joint initiative between the department “Scale bridging thermodynamic and kinetic simulation” at ICAMS and the CM. *Comp. Mat. Sci* 108, 293 (2015).



Activities within the Cluster of Excellence “Ruhr Explores Solvation” (Resolv) at MPIE



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M. Stratmann ¹, J. Neugebauer ²

¹ Department of Interface Chemistry and Surface Engineering (GO)

² Department of Computational Materials Design (CM)

The Cluster of Excellence Resolv was established at the Ruhr-Universität Bochum (RUB) in November 2012, and is led by Prof. Dr. Martina Havenith-Newen (Physical Chemistry 2) as speaker of the cluster. Besides several departments from RUB, groups from TU Dortmund, University Duisburg-Essen, Fraunhofer UMSICHT Oberhausen, MPI für Kohlenforschung (Mülheim a. d. Ruhr), MPI for Chemical Energy Conversion (Mülheim a. d. Ruhr) and MPIE, are members of the cluster. The mission of Resolv is to establish an understanding of solvation processes at a molecular level. The cluster is structured in 3 research areas: (A) Understanding and Exploiting Solvation in Chemical Processes, (B) Connecting Solvation Dynamics with Biomolecular Function and (C) Ion Solvation and Charge Transfer at Interfaces. With their expertise in interface science, two departments GO and CM) of MPIE are strongly involved in research area C. MPIE is also involved in the coordination of the cross-linking topic “ion hydration”.

So far, 5 doctoral students started their work at MPIE within the cluster’s Graduate School of Solvation Science (GSS). Their projects involve

surface force measurements on solvated well-defined alloy systems, preparation and investigation of “emersed electrodes”, vibrational spectroscopy of solvent modes as function of electrode potential, simulation of solvated ions by *ab initio* molecular dynamics, and experimental characterisation of the defect-formation in zinc oxide. The MPIE is also involved in a number of collaborative projects of students based at RUB. Most projects with MPIE involvement call for a strong collaboration between experimentalists and theorists.

Initial results of the projects based at MPIE include investigations of the stability of polar ZnO(0001) surfaces in an aqueous environment via construction of surface Pourbaix diagrams, an accurate description of ions in solution, the setup of an experiment that enables emersion of well-defined electrodes from an electrolyte under control of the electrode potential with direct transfer to a UHV chamber, and the characterisation of the potential-dependent desolvation of germanium after an electrochemically triggered surface termination change.

More details: <http://www.rub.de/solvation>



Fig. 1: Research areas within RESOLV ((C) Ruhr-Universität Bochum).



Max Planck Research Group on High Temperature Materials

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²previously: Department of Structure and Nano-/Micromechanics of Materials (SN)

³Alloy Design, Ruhr-Universität Bochum

Since May 2015, Gunther Eggeler, professor for materials science at the Ruhr-Universität Bochum (RUB), leads a new Max Planck Research Group on high temperature materials (HTM), which emanated from the Max Planck Fellow Group which existed from May 2010 until May 2015. The new HTM group seamlessly continues the research activities in the area of high temperature materials. Aleksander Kostka, a scientific member of the previous Max Planck Fellow Group, is now a transmission electron microscope (TEM) scientist at the Ruhr-Universität Bochum. This position allows him to also keep an involvement in the new high temperature materials group. The expertise of the group lies in the field of exploring and explaining microstructural evolution

during processing and service of engineering materials. Recently the group has contributed to a better understanding of the nucleation of the Mo-rich Laves phase during aging and creep of tempered martensite ferritic steels (TMFS) [1, 2]. It could be shown that segregation of minor alloy elements like Si to micro grain boundaries precedes the nucleation of Laves phase particles [1, 2]. As an example, Figures 1a and b show that TEM and 3D atom probe (APT) investigations (Department of Microstructure Physics and Alloy Design, MPIE) yield complementary information which is needed to identify elementary transformation and deformation processes which govern creep.

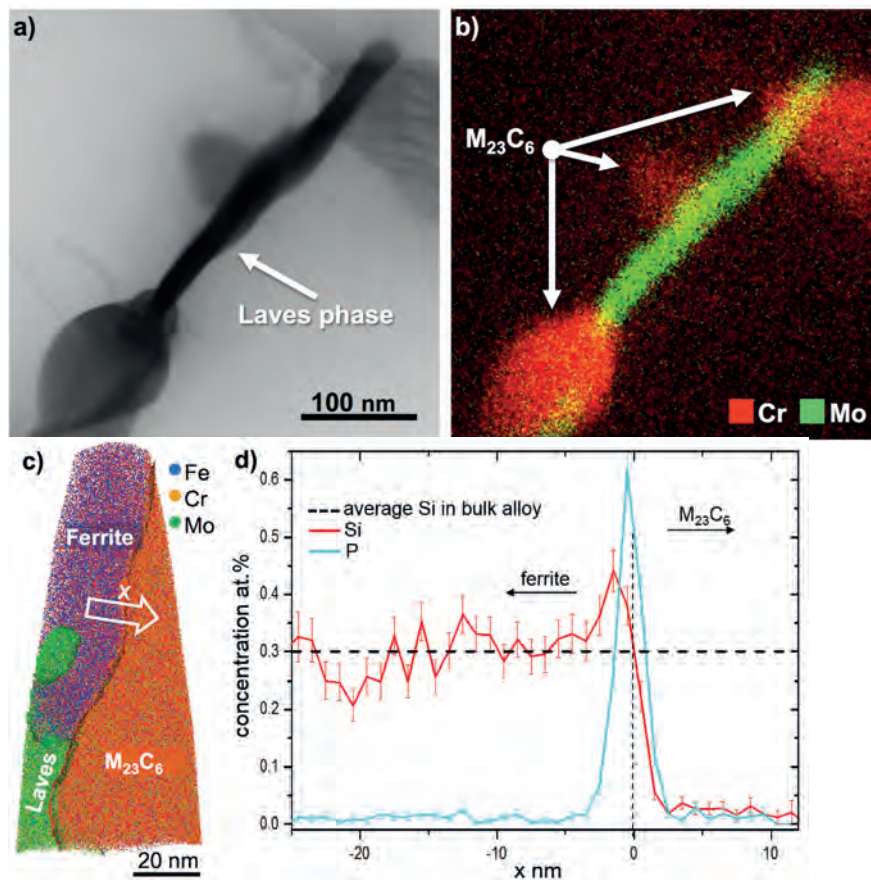


Fig. 1: TEM/APT analyses of a 12%Cr tempered martensite ferritic steel after 82.000 hours of creep at 650°C, 120MPa. (a) Scanning TEM (STEM) bright field image showing a small Laves phase along the grain boundary. (b) Corresponding energy-dispersive X-ray spectroscopy (EDX/EDS) map of Cr (orange, highlighting carbides) and Mo (green, denoting a Laves phase). (c) APT reconstruction showing the distribution of atoms (Fe, blue; Cr, orange; Mo, green). Violet, orange and green regions denote ferrite, $M_{23}C_6$ and Laves phase, respectively. White arrow marks the positions of the concentration profile plotted in (d). (d) Concentration profile of Si and P along x axis.

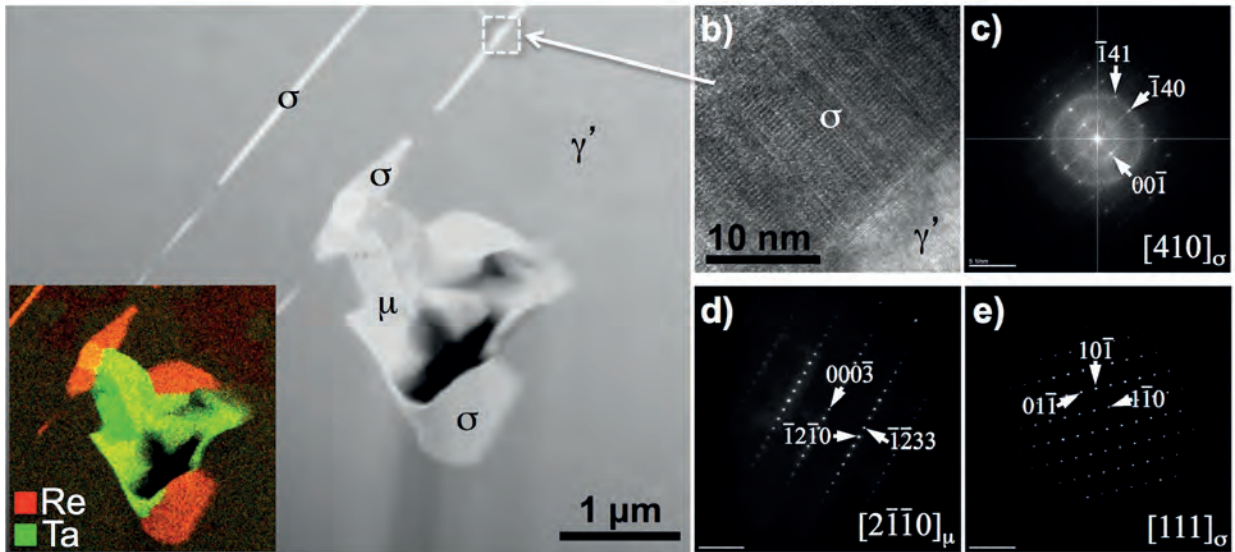


Fig. 2: TEM analysis of precipitations in Ni-base superalloy ERBO/1 after 1000°C, 32 h heat treatment. a) STEM HAADF (high-angle annular dark field) micrograph with an EDS inset showing the distributions of Re and Ta; b) High resolution TEM (HRTEM) micrograph of the needle-like σ -phase precipitate and c) corresponding FFT; d) and e) characteristic selected area diffraction patterns taken from σ and μ -phase, respectively.

The HTM group also performs research in the area of single crystal Ni- and Co-based superalloys. The HTM group organized collaboration between several partners which yielded a full microstructural characterization of a Ni-base superalloy, addressing macroscopic, mesoscopic and atomistic phenomena [3]. There was also an effort to study the thermodynamics and kinetics of TCP phase formation during high temperature exposure [4]. As an example, Fig. 2 shows a TEM analysis of precipitations in Ni-base superalloy ERBO/1 after 1000°C, 32 h heat treatment where σ - and μ -phase are clearly distinguishable. The needle-like σ -phase forms initially in the $\{111\}$ γ -phase habit planes as can be observed in Fig. 2a or more clearly in Fig. 2b and c. The σ -phase consists of Cr, Mo and small fraction of Re, while μ -phase - mainly of Hf and Ta. At 1100°C the σ -phase seems to be not stable: precipitates are found only after short heat treatments while they disappear after 50 h annealing time. At 1200°C, no precipitates of the σ -phase form at all, regardless of the annealing time.

In the superalloy field, HTM acts as a link between MPIE researchers and materials researchers at other affiliations (Universities of Bochum and Erlangen, Deutsches Luft- und Raumfahrtzentrum and Forschungszentrum Jülich). For the future it is

planned to continue the successful collaboration with the MPIE departments of Dierk Raabe and Gerhard Dehm, combining the group's expertise in high temperature materials with *in-situ* TEM experiments (Dehm department) and the 3D atom probe (Raabe department). Emphasis will be placed on the improvement of existing and the development of new high temperature materials. Moreover, new advanced topics like high temperature shape memory alloys, high entropy alloys and microstructural evolution during additive manufacturing will be tackled.

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Research Funding

K. Hübel

Professional Support in Research Funding

In the reporting period 2013-2015 Dr. Katja Hübel provided professional support in national, European and international research funding to the scientists of the MPIE with the aim of increasing the success of funding applications. She started working at the MPIE in February 2013 and gave advice on all kinds of funding from individual (e.g. Humboldt Research Fellowship for Postdoctoral Researchers, Marie Skłodowska-Curie Individual Fellowship and ERC Grants) to project funding (e.g. Horizon 2020, German Ministry for Science & Education and German Research Foundation). Furthermore special attention was drawn to prizes and awards (e.g. Leopoldina Preis, Adolf-Martens-Preis and deutscher Studienpreis). Support in research funding was oriented on the strategic development of the MPIE as well as on its interdisciplinary research focus.

Competitive Applications

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

Project Management

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

Support for Junior Scientists

In the current reporting period 2013-2015 a special focus was set on young scientists. This included not only finding the right funding scheme that fits to their scientific profiles but also strategic advice for their career development. Furthermore a workshop "Competitive Proposal Writing" has been given for the first time in summer 2015 and will be offered in yearly intervals. Another workshop "Applying for ERC Starting & Consolidator Grants" is organized yearly with the MPI for Radioastronomy in Bonn and with the National Contact Point ERC. This workshop is a special offer to junior scientists at Max Planck Institutes in North Rhine-Westphalia. Support was

given to junior scientists working at the MPIE as well as to prospective junior scientists planning to work at the MPIE.



Fig. 1: Dr. Pascal Beese (right) received the Leopoldina Prize for Junior Scientists. Left: Prof. Jörg Hacker, President of Leopoldina. © Christof Rieken, Leopoldina

Selected Research Funding

Dr. Blazej Grabowski got an *ERC Starting Grant of the European Research Council*, Horizon 2020, "Time-scale bridging potentials for realistic molecular dynamics simulations-TIME-BRIDGE", ERC Jul 2015 – Jun 2020.

Dr. Markus Valtiner got the „*Max-Buchner-Forschungsstipendium*“ starting in Jul 2014

Dr. Karl Mayrhofer won the *Dechema-Preis der Max-Buchner-Forschungstiftung*, Nov 2014

Christian Broß won the *Apprentice Prize of the Max Planck Society*, Jul 2015.

"International Max Research School (IMPRS) for Surface and Interface Engineering in Advanced Materials (SurMat)", Follow-up application, *Max Planck Society*, Jan 2016 – Dec 2020.

"Enhanced substrates and GaN pilot lines enabling compact power applications – PowerBase", *The ECSEL Joint Undertaking, FP7*, May 2015 – Apr 2018.

Dr. Raheleh Hadian (Iran), McMaster University Hamilton/Canada, *Humboldt Research Fellowship for Postdoctoral Researchers*; Mar 2015 to Feb 2017.



PhD Representatives

C. Baron, P. Bhogireddy, V. Dandapani, S. Hieke, Z. Li, W. Luo, A. Wengert

The PhD representatives are the interdepartmental representation of the PhD students at the Max-Planck-Institut für Eisenforschung (MPIE) GmbH. All PhD students at the MPIE elect once a year seven representatives, one or two per department. The target of the representatives is to maintain and further develop the interdisciplinary cooperation and exchange of PhD students. They also serve as connection to the PhDnet, a platform for exchange among doctoral students of all Max Planck Institutes and provide their fellow colleagues at the MPIE with up-to-date information about stipend and contract possibilities, insurance information and other upcoming topics.

Further, the PhD representatives initiate and organize soft skill courses for PhD students on a regular basis with topics such as scientific writing, presentation and leadership skills. Additionally, career talks are organized to give PhD and postdoctoral students a chance to build up industrial contacts and to get an insight about job possibilities in different industrial branches. In 2015 career talks with



Fig. 1: Career talk with BASF on 18 November 2015.

the participation of the companies ThyssenKrupp Steel Europe, McKinsey and BASF received highly positive feedback. With the goal of improved knowledge transfer in the Institute, the representatives also launched a lecture series in 2015 on important scientific tools and techniques which are of general interest to them. Examples were introduction to Matlab and electron backscatter diffraction. The events and courses received a lot of positive response from the PhD students in the Institute and will be continued.



Fig. 2: Networking at the first career talk held at the MPIE with guest speakers from ThyssenKrupp Steel Europe.



Alumni Networking

B. Kohlhaas

Working at the MPIE has significantly influenced the career of many scientists. Over the years, a considerable number of employees have worked at the Institute in various fields and departments. To encourage a lively exchange among the former Institute members, the alumni network has been established in our Institute in 2003 and has 300 members at present.

What does “Alumni” mean?

The word “alumnus” is derived from the Latin “alere”, “to nourish”, and means “nursling” or “pupil”. “Alumni”, the plural form of “alumnus”, literally means “the nurtured ones”. In the USA, the term “alumni” has been used for graduates or former students of institutes of higher education for more than 200 years. In Germany, it is only since the last few years that alumni activities are being established at universities and research institutes. (www.wikipedia.de)

Who are Alumni?

Alumni are all former staff members of the MPIE who worked at the Institute for more than six months, regardless of whether they worked here as a scientist or technician, in the workshops, library, or administration.

What are the aims of the Alumni Network?

The Network cultivates contact and shares experiences. A major intention is to establish a

platform for discussions of common interests and problems, and for sharing both professional and personal experiences.

Keep in Touch

The alumni network will help not only to stay in contact with former colleagues, but also to forge new links with current Institute members, thus supporting an active and beneficial exchange for all.

Members of the alumni network will be kept updated about news, events and actions of the MPIE by mail or e-mail.

Special alumni meetings with scientific lectures and a full social programme allow members to meet colleagues, share experiences and forge ties that may lead to new collaborations.

In addition, you can inform yourself about Alumni News and easily join to the Network online <https://alumni.mpie.de>

Alumni Meeting 2014

Our last Alumni Meeting, “Applied fundamental research for nearly 100 years”, took place at MPIE on 28 May 2014 on the occasion of the 60th Birthday of Prof. Martin Stratmann.

180 guests, including alumni and current MPIE employees, attended the successful event.



Fig. 1: Networking at the Alumni Meeting 2014.



Fig. 2: Prof. M. Stratmann giving a speech at the Alumni Meeting 2014.



The Institute in Public

Y. Ahmed Salem

Public Relations

The reporting period 2013-2015 was full of events and changes for the Public Relations (PR) of the MPIE. A communication strategy with defined target groups and a portfolio of different methods and measures to reach the defined target was developed. The current reporting period was characterized by the implementation of the different measures. Here, the PR mainly concentrated on two sections: the "traditional" press relations on the one hand including a website relaunch in May 2015, and event management on the other hand.

Event Management

Besides various tours through the Institute and its laboratories for pupils, students, interested groups out of the broad public and for politicians and industrial partners, some major events took place in the years 2013-2015. Some of them will be presented in the following:

Science Days at the Theodor Fliedner School, February 2013 and 2014

Once a year the Theodor Fliedner School organizes the so-called Science Days where companies and institutions either visit the school and present their work or invite interested pupils to their facilities. The PR regularly takes part in this event and either organizes lab tours through the Institute or trains doctoral students to give an interactive talk with small experiments to the pupils at the school. The talks in 2013 and 2014 were held by Tom Jäpel and Emanuel Welsch, doctoral students in the department Microstructure Physics and Alloy Design (MA).

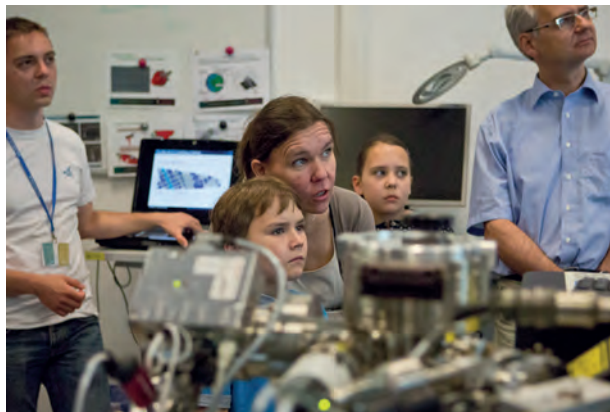


Fig. 1: *In the laboratory of the atom probe during the Open Day 2013.*

FameLab, March 2013

Fady Archie, previously master student and now doctoral student in the MA, was coached by the PR to participate at the FameLab 2013, a science slam competition. Archie won the competition for North Rhine-Westphalia and had the chance to take part at the competition in Berlin, where all winners of the German states took part. There he made the second place.



Fig. 2: *Dr. Markus Valtiner, group leader in the Department of Interface Chemistry and Surface Engineering (GO), while explaining his research during the Researchers' Night 2013 in Düsseldorf.*

Open Day, July 2013

Together with the Steel Institute VDEh, the MPIE organized an Open Day in the summer of 2013 which attracted over 2500 people. The programme comprised over 30 different MPIE attractions showing the research in different ways to young and old.

Düsseldorfer Tage der Berufsorientierung – Days of Career Orientation, July 2013

The career orientation days in Düsseldorf were organized mainly by the City of Düsseldorf and the Chamber of Commerce and financially supported by the European Union and the Federal Ministry of Labour and Social Affairs. Pupils had the chance to spend one day at the MPIE and get an insight into the different research areas as well as the various apprenticeship possibilities at the Institute.



Researchers' Night in Düsseldorf, September 2013 and 2015

The first Researcher's Night in Düsseldorf was organized 2013 by the Heinrich Heine University Düsseldorf and the University of Applied Science Düsseldorf and took place in various locations all over the city. The PR organized the MPIE's participation with attractions in three fields: energy and environment; technology and medicine & technology and society. The Researchers' Night 2015 took place in the heart of the City Düsseldorf, where the PR and doctoral students of different departments presented research results on TiNb hip implants and additive manufacturing, to a broader public.

MS Wissenschaft, Mai – September 2014

In 2014 the MPIE took part at the MS Wissenschaft, an exhibition ship which serves as a floating science centre and cruised from May till September 2014 in around 40 cities in Germany and Austria. This exhibition is supported by the Federal Ministry of Education and Research. Its topics match the current Science Year's topic which is proclaimed by the Ministry. The "Ageing Society" was the central theme in 2014, where the PR presented the work of the department of Computational Materials Design (CM) on TiNb hip implants and on computer simulations being an important pillar in modern alloy design.

KopfSalat, since September 2014

KopfSalat is a new event taking place twice a year at the Institute and inviting researchers of different research areas to give generally understandable talks to the broad public of Düsseldorf. The intention is not to give an insight on the MPIE research but to attract people to come to the Institute by offering different research topics. The PR invited Prof. Dr. Martin Wikelski from the Max Planck Institute for Ornithology, Radolfzell, as the guest speaker of the first KopfSalat event in September 2014. Prof. Wikelski explained how animal migration can be used as a warning system for natural catastrophes and how he is working to build up a satellite supported



Fig. 3: KopfSalat invites twice a year researchers of different research areas to give generally understandable talks at the MPIE. This is an impression of the second event in April 2015.

system to do so. This event attracted around 70 visitors who entirely subscribed to the PR mailing list to be informed regularly about public events taking place at the MPIE.

The second KopfSalat event attracted about 92 visitors and was led by Prof. Hanns Hatt, professor and researcher in the field of cell physiology at the Ruhr-Universität Bochum. He explained how smelling functions, how this is influenced by hormones and how everyone can influence other people's perception by using different odours.

The third KopfSalat was at the end of November 2015. This time Prof. Gerd Gigerenzer from the Max Planck Institute for Human Development, Berlin, was invited to give a talk on adaptive behaviour and cognition. The event was visited by 120 persons.

The PR chooses the various speakers mainly out of the winners of the Communicator Prize of the German Research Foundation. This prize is awarded once a year to researchers who are especially able to present their work in a generally understandable and entertaining way. To attract as many visitors



Fig. 4: Sample Preparation at the Girls' Day 2015.

as possible to KopfSalat, the event is announced in the local newspaper, different online and social media sites and through post cards which are distributed in the neighbourhood of the Institute, at the Heinrich Heine University Düsseldorf and at the Adult Education Centre Düsseldorf.

Girls' Day, April 2015

A group of girls had the chance to get an insight on the work of a Max Planck scientist on 23rd April 2015, the nationwide Girls' Day. At the Girls' Day companies, universities and different organizations invite female pupils once per year for one day. The girls learn about possible trainings and courses of study in fields usually dominated by men like IT, craft, natural sciences and engineering. The girls were supervised by doctoral students of the Max Planck Research Group Nanoanalytics and Interfaces and examined different samples with light microscopes and a scanning electron microscope.

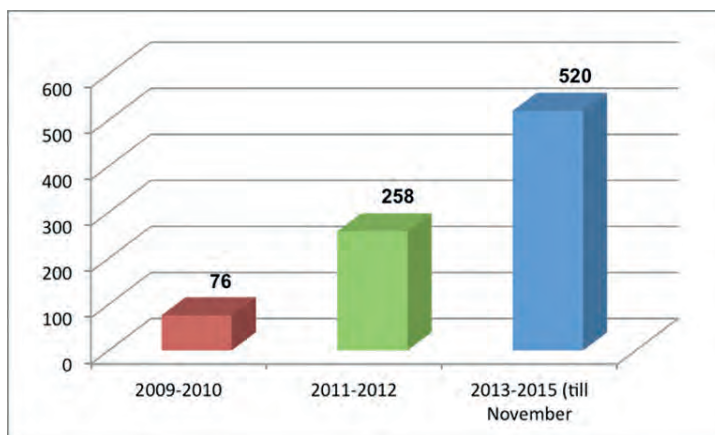


Fig. 5: Comparison of absolute amount of clippings during different reporting periods.

Press Relations

The Institute was mentioned 520 times in newspapers/journals and websites and 39 press releases were written during the reporting period (till 1st of November). These are more than four times more clippings compared to 2009-2010 and a plus of 25% compared to the last reporting period 2011-2012.

Selected articles published in different print and online newspapers and journals:

20th October 2013 Frankfurter Allgemeine Sonntagszeitung: Damit er nicht so schnell kaputtgeht

27th November 2013 Rheinische Post: Düsseldorfer Forschung führt in die ganze Welt

17th March 2014 Welt der Physik: Reale Materialien aus dem Computer

5th November 2014 Die Welt: Atomsonden erforschen Nanostrukturen

5th February 2015 Spiegel Online: Hochfest und Leicht – Nanokristalle formen den Superstahl

23rd March 2015 Springer Professional: Symphonie im Stahl

The **MPIE newsletter** as a specific tool to reach the industrial partners and other publics interested in materials science, was restructured. Compared to former editions which covered different topics per release, the newsletter now focuses on one topic each, as e.g. the edition 01/2015 which is about hydrogen embrittlement. This way important topics of the Institute's interdepartmental research are highlighted and the interdepartmental research activities are better shown.

The **website of the MPIE** was relaunched in May 2015 including a complete new backend system, layout and structure. The layout is based on the corporate identity of the Max Planck Society to have a better brand recognition. The website is now

structured to reach the different target groups of the Institute like scientists but also industrial partners, journalists and the broad public. Therefore, it contains all basic information both in German and in English.



Fig. 6: The MS Wissenschaft is a floating science centre where the MPIE presented how simulations can help in developing better hip implants. Copyright: Ilja Hendel, Wissenschaft im Dialog

Internal communication and new communication tools

The MPIE Intranet was further developed by adding information on child care, business travel, living in Germany, funding and events taking place in and outside the MPIE. It is now a first address especially for newcomers to inform themselves about internal matters. In the future it will also contain all regularly required forms and templates like flexitime sheets.

The PR has also designed a MPIE calendar with scientific eye-catching images as a new marketing and communication tool. Since 2014 the calendar is distributed to industrial partners, journalists and visitors. It gives an impression on the variety of research topics at the Institute.