PART I.

THE INSTITUTE

Management of the Institute

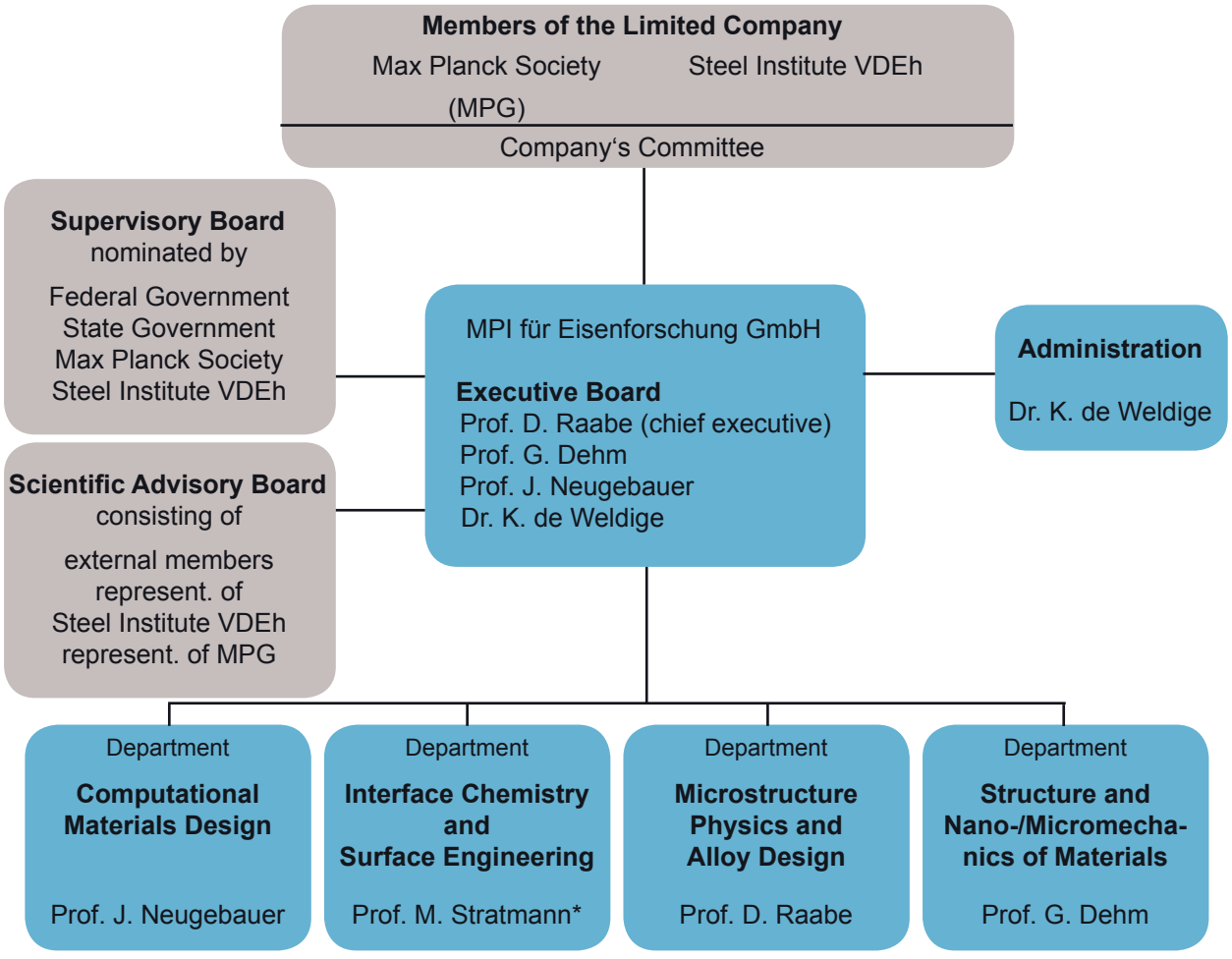
The Max-Planck-Institut für Eisenforschung GmbH (MPIE) is a joint venture between the Max Planck Society and the Steel Institute VDEh. As the Institute's budget is supplied indirectly through industry, this Institute is unique within the Max Planck Society.

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 head 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, the Supervisory Board, reflects and debates with the MPIE management and shareholders on strategic questions regarding fundamental scientific research related to corporate purpose; it 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. 2018)



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

Scientific Organization

The Institute conducts fundamental research on metallic alloys and related materials. It pursues a holistic approach where material systems are studied under consideration of their complex compositions and nanostructures on the one hand and their exposure to extreme environmental conditions on the other hand.

The Institute has four departments:

- Computational Materials Design (Prof. J. Neugebauer): exploration of materials properties and processing based on *ab initio* (parameter free) multiscale simulation techniques
- Interface Chemistry and Surface Engineering (Prof. M. Stratmann; on leave, provisional head: Prof. J. Neugebauer): environmentally accelerated degradation of surfaces and interfaces by corrosion and de-adhesion. Engineering of new and stable surfaces and interfaces
- Microstructure Physics and Alloy Design (Prof. D. Raabe): Development of novel metallic materials and microstructures with improved mechanical and functional properties using simulation, microscopy, atom probe tomography and diffraction methods
- Structure and Nano-/Micromechanics of Materials (Prof. G. Dehm): Understanding the structure and imperfections of metals, alloys and intermetallics by advanced electron microscopy and exploring their interplay with mechanical and tribological properties at small length scales by *in situ* nano-/micromechanical approaches

Each department consists of several research groups, which are managed by group leaders. In addition to the departments an Independent Max Planck Research Group, headed by Prof. C. Scheu on Nanoanalytics and Interfaces has been established in 2014. The figure on page 12 shows the organization of the groups and their scientific aims. Part II of this report reviews the scientific concepts and achievements of the departments and the research conducted in the individual groups.

In addition to the individual scientific goals of the departments, many research topics are highly interdisciplinary and require the experimental and theoretical expertise of several departments, enabling breakthroughs in highly competitive research areas. Part III describes selected scientific highlights including such interdepartmental research activities. It is divided into the four topics:

- Stability of Surfaces and Interfaces
- Microstructure-related Materials Properties
- Development of Advanced Materials
- Scale-bridging Simulation and Materials Informatics

Several short papers on selected scientific topics provide an overview of results obtained during the last years for each of these central research areas.

The organisation of research within the MPIE, both vertically in departments and research groups with highly specialized expertise as well as horizontally in the form of interdepartmental research activities, combines a high level of individual scientific skills available within each department and group with a highly ambitious cross-disciplinary spirit among them. It is the basis for the development of advanced materials with useful properties, combining, e.g., high mechanical strength with high surface functionality. This approach unleashes very high scientific synergy effects, which have enabled several exciting discoveries, more efficient use of scientific equipment, a homogeneous research profile and intense interdepartmental collaborations.

Service groups support the scientific departments with valuable expertise. These include the production, processing and testing of materials, chemical analysis of metallic substrates, metallography and a mechanical workshop equipped for handling hard and brittle materials, facilities to design and 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

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
Stability of Surfaces and Interfaces			
Microstructure-related Materials Properties			
Development of Advanced Materials			
Scale-bridging Simulation and Materials Informatics			

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

Scientific Groups and Departments (2018)

Computational Materials Design
Prof. J. Neugebauer

- **Adaptive Structural Materials** — Dr. B. Grabowski
- **Computational Phase Studies** — Dr. T. Hickel
- **Computer Centre** — Dr. C. Freysoldt
- **Defect Chemistry and Spectroscopy** — Dr. C. Freysoldt
- **Electrochemistry and Corrosion** — Dr. M. Todorova
- ERC Starting Grant **Bridging Length and Time with Atomistic Simulation** — Dr. B. Grabowski
- Fund. by The Netherlands Organisation for Scientific Research **Complex Concentrated Alloys** — Dr. F. Körmann (since Mar. 2017)

Interface Chemistry and Surface Engineering
Prof. M. Stratmann*

- **Atomistic Modelling** — Dr. S. Wippermann
- **Corrosion** — Dr. M. Rohwerder
- Guest Group **Electrocatalysis** — Dr. K. J. J. Mayrhofer
- Guest Group **Interaction Forces and Functional Materials** — Dr. M. Valtiner
- Guest Group **Interface Spectroscopy** — Dr. A. Erbe

Microstructure Physics and Alloy Design
Prof. D. Raabe

- **Adaptive Structural Materials** — Dr. C. Tasan (until Dec. 2015)
- **Alloys for Additive Manufacturing** — Dr. E. Jägle
- **Atom Probe Tomography** — Dr. B. Gault (since Jan. 2016)
- **Biological Composites** — Dr. H. Fabritius (until Dec. 2017)
- **Combinatorial Metallurgy and Processing** — Dr. H. Springer
- **Microscopy and Diffraction** — Dr. S. Zaefferer
- **Mechanism-based Alloy Design** — Dr. D. Ponge
- **Theory and Simulation** — Dr. F. Roters
- ERC Consol.Grant **Hydrogen in Energy Materials** — Dr. B. Gault (since Jan. 2018)
- Funded by German Research Foundation **High-Entropy Alloys** — Dr. Z. Li (since Jan. 2017)
- Funded by BMBF **Materials Science of Mechanical Contacts** — Dr. M. Herbig (since Oct. 2016)
- Joint group with RWTH Aachen **Interface Design in Solar Cells** — Dr. O. Cojocaru-Mirédin

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

- **Advanced Transmission Electron Microscopy** — Dr. C. Liebscher
- **Intermetallic Materials** — Dr. M. Palm, Dr. F. Stein
- **Nano-/ Micromechanics of Materials** — Dr. C. Kirchlechner
- **Nanotribology** — Dr. S. Brinckmann
- **Thin Films and Nanostructured Materials** — Dr. M. Ghidelli (since Nov. 2018)
- Max Planck Research Group **High Temperature Materials** — Prof. G. Eggeler

Independent Max Planck Research Groups

- **Nanoanalytics and Interfaces** — Prof. C. Scheu
- **External Members and Fellows**
- Max Planck Fellow **Self-Reporting Materials** — Prof. J. Schneider
- External Scientific Members — Prof. M. Hillert, Prof. R. Kirchheim

Scientific Service Groups

- **Synthesis and Processing** — Alloy production, Annealing & welding, Thermom. treatments — Dr. H. Springer
- **Metallography** — Light microscopy, Metallographic sample preparation, SEM — Dr. S. Zaefferer
- **Materials Testing** — Mechanical properties, Physical properties, Varying environments — Dr. H. Springer
- **Research Coordination Office** — Doctoral Programme, International Office, Public Relations, Research Funding — Dr. K. Hübel

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

Recent Developments

Here we reflect some of the recent changes at the MPIE between 2016 and 2018 which have helped us to strengthen the Institute's scientific profile and administrative efficiency. For evaluation purposes some trends are described over the past 6 years.

Constructions on G. Dehm's advanced electron microscopy laboratory which is a key facility of his department on "Structure and Nano-/ Micromechanics of Materials" have been finished and a scientific opening workshop was held in November 2018. The microscopy group is a key asset in many cross-departmental research projects including interfacial interaction phenomena of structure and composition effects, precipitation and transformation phenomena and high entropy alloys as well as in essential methodological projects related particularly to the correlative use of electron microscopy and atom probe tomography. Activities by the department are further boosted by the ERC Advanced Grant that has been awarded to G. Dehm in 2018.

Likewise the independent research group on "Nanoanalytics and Interfaces" which was opened by C. Scheu in 2014 is fully operative, pursuing multiple interdisciplinary collaborations in the fields of hydrogen generation and fuel cell related materials problems, materials for catalysis and nanostructured thermoelectrics.

Permanent scientific stimulation among in-house groups and departments but also with external partners takes place through a bi-weekly inter-departmental workshop series where topics of high mutual interest are identified and discussed. In these discussion rounds often younger scientists take the lead and initiate new collaborations or identify new promising joint research topics of high scientific risk, gain and synergy. Examples for topics discussed in recent months are high throughput experiments, complexions, big data and machine learning in microscopy and atom probe tomography, simulation-enhanced analytical field ion microscopy, grain boundary segregation and ordering effects, high strength aluminium alloys, defect phase diagrams, medium and high entropy alloys, magnetic high entropy alloys, thermodynamics of high entropy alloys, nanostructures in tribological interfaces, lattice defects in thermoelectric materials to name a few.

Over the past 6 years several new research groups have been initiated, namely, the group on "Nanotribology" by S. Brinckmann, the group for "Nano-/Micromechanics of Materials" by C. Kirchlechner, the group for "Advanced Microstructure Characterization" by C. Liebscher, the group on "Electrochemistry and Corrosion" by M.Todorova and the group of "Synthesis of Thin Films and Nanostructured Materials" by M. Ghidelli (since November 2018). Further groups have been set up with the support of third-party funding, namely, the BMBF-funded group on "Interface Design in Solar Cells" by O. Cojocaru-Mirédin who is now a group leader at RWTH Aachen and co-operates the group under the new designation "Advanced Functional Materials", a BMBF NanoMatFutur funded group on "Semiconducting nanocomposites with tailored electronic and optical properties for solar energy conversion" by S. Wippermann, a BMBF funded group by M. Herbig on "Materials Science of Mechanical Contacts", a DFG supported initiative on "High-Entropy Alloys" headed by Z. Li, a group funded by the joint Max-Planck-Fraunhofer initiative on "Alloys for Additive Manufacturing" by E. Jägle, an ERC Starting Grant group by B. Grabowski on "Bridging Length and Time with Atomistic Simulation", a group on "Hydrogen in Energy Materials" which is funded by an ERC Consolidator Grant awarded in 2018 to B. Gault, and a group funded by the Netherlands Organisation for Scientific Research on "Complex concentrated Alloys" headed by F. Körmann. Guest groups have been set up in cooperation with our former group leaders: a guest group on "Electrocatalysis" by K.J.J. Mayrhofer (now at Helmholtz Institute Erlangen-Nürnberg for Renewable Energy), a guest group on "Interaction Forces and Functional Materials" by M. Valtiner (now at TU Vienna) and a guest group on "Interface Spectroscopy" by A. Erbe (now at NTNU Trondheim).

Also, several large-scale and networking projects were initiated or prolonged during the last years. Examples are European research networks like "AccMet - Accelerated Metallurgy - the accelerated discovery of alloy formulations using combinatorial principles", „ADVANCE: Sophisticated experiments and optimisation to advance an existing CALPHAD database for next generation TiAl alloys", "EPPL - Enhanced Power Pilot Line", „HERCULES-2 - Fuel flexible, near-zero emissions, adaptive performance marine engine", "MECANO - Mechanics of Nano-Objects", "PowerBase - Enhanced substrates and GaN pilot lines enabling compact power applications" as well as currently 6 RFCS projects. Another important collaboration is the cooperation 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 included the joint group on "Adaptive Structural Materials" which was funded through an ERC Advanced Grant that was jointly awarded to J. Neugebauer and D. Raabe for the development of novel experimental and theoretical tools to

understand mechanisms associated with metastable phases in structural alloys. A newly established Max-Planck network project entitled BigMax is concerned with Big Data and Machine Learning in materials science and engineering (https://www.bigmax.mpg.de). It is an initiative where 10 Max Planck institutes collaborate on big data, machine learning, software science, applied mathematics and materials science. The MPIE is also participating in the MaxNet Energy, a research cooperation that aims at obtaining a deeper understanding of fundamental processes in chemical energy conversion and the rational design of novel catalysts, materials and techniques. Further funded networks include RESOLV (DFG Excellence Cluster), Initial Wear (Max-Planck-Fraunhofer initiative), CarMon (Leibniz-Max-Planck initiative) and the "Knowledge and Technology Platform for Prediction of Durability and Lifetime of Organic Coated Metals under Long-Term Environmental Corrosion" (joint project with the Free University of Brussels (VUB), within the framework of the Research Foundation - Flanders (FWO).

The Institute pursues also several large joint initiatives with its neighbour universities RWTH Aachen (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 S. Wippermann; 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. Further joint collaborative research centers funded by Deutsche Forschungsgemeinschaft are the SFB TR 188 (damage controlled forming) with University Dortmund and SFB 1232 (combinatorial alloys design) with Bremen University.

Over the past years several group leaders from the MPIE received professorship offers from prestigious universities such as Ohio State University, Delft University, University Bern, Trondheim University, Erlangen University, University of Vienna, RWTH Aachen, University of Stuttgart, Massachusetts Institute of Technology (MIT), Shanghai Jia Tong University, Xian University, Indian Institute of Technology Bombay, Postech and the Korea Institute of Science and Technology KIST.

MPIE members were and are also involved in several science community und 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), as head of the division Metals and Materials of the German Physical Society DPG (J. Neugebauer), member of the selection committee for the allocation of Humboldt Research Fellowships (G. Dehm), member of the selection committee for Max Planck Research Groups and the Lise Meitner Excellence Program (G. Dehm), panel member for the strategic evaluation of the research field "Matter" of the Helmholtz Association of German Research Centres (C. Scheu), member of the evaluation board of the state North Rhine Westphalia "EFRE NRW 2014 – 2020 "Neue Werkstoffe NRW" (C. Scheu), as a member of the German Council of Science and Humanities (Wissenschaftsrat) (D. Raabe), the Joint Strategy Commission (Strategiekommission) for the German Excellence Initiative (D. Raabe) and the Senat of the Helmholtz Association of German Research Centres (D. Raabe).

Several highly visible awards were won by MPIE members in the past years, among them an ERC Advanced Grant, an ERC Co-Investigator Grant, an ERC Consolidator Grant and two ERC Starting Grants, 6 Alexander von Humboldt Awards for Senior Professors, one Otto-Hahn Medal, the NRW Innovation Award, the DECHEMA-Award and 3 BMBF Junior Research Group Awards to name a few important recognitions. In 2017 the MPIE reached the 3rd best position in the Humboldt-Ranking among all non-university research organizations in Germany with 30 scholarships won between 2012 and 2016.

In order to stay connected to graduates and former scientists of the Institute and to benefit from their experience MPIE established a very active alumni network. A website launched in 2015 presents the Institute's research and achievements to different target groups ranging from scientists over students, journalists, politicians, industry representatives and pupils to the wider public. Measures to increase the Institute's visibility and to attract the next generation of junior scientists are: a new series of lectures named "KopfSalat", regular Kids' Labs, Kids' Universities and participation in the Girls' Day.

In the last years several measures to reconcile the demands of family and career have been taken. Since 2017 we have four reserved places in a day-care facility for children, in 2018 a Gender Equality Officer was elected and the Institute appointed a "MPIE Female Science Career Mentor".

With all these recent achievements we look now upon a period of more than 100 years of successful and sustainable public private partnership between the Max Planck Society and the European industry. We celebrated this anniversary in 2017 with a scientific colloquium, a public outreach program and a scientific project reviewing the history of the Institute.

Max Planck Research Groups

Independent Max Planck Research Group on Nanoanalytics and Interfaces

Group Head: C. Scheu

Research Mission and Scientific Concepts

The scientific mission of the independent research group Nanoanalytics and Interfaces (NG) is to explore materials in all aspects of renewable energy applications by in-depth analyses of their nano-/microstructure. Material science concepts are less frequently applied in this field despite of their importance to design materials with better performance. The NG group correlates the findings of the nano-/microstructural analysis to the growth conditions and functionality, and uses them to improve synthesis and processing strategies. A strong focus is laid to understand the stability of nanostructured materials in harsh environments such as elevated temperatures.

Fig. 1: *The NG group in summer 2018.*

The core expertise of the NG group are *ex situ* and *in situ* electron microscopy-based techniques including aberration-corrected (scanning) transmission electron microscopy ((S)TEM), electron energy-loss spectroscopy (EELS), energy-dispersive X-ray spectroscopy (EDX), *in situ* heating experiments and electron tomography [1-25].

The NG group was founded in April 2014 by C. Scheu, who is at the same time a full professor at the RWTH Aachen University. The group has bachelor and master students, PhD candidates, and postdocs (Fig. 1), and a strong commitment in training and scientific education of young professionals. The group is also actively involved in supervising summer students and pupils of the Girl's day. Seven PhD students, two master students, and one bachelor student have graduated from the NG group with great success since 2014, some of them with distinction. Besides teaching at the RWTH Aachen University, C. Scheu also actively participates as a committee member in several university boards. She is also active as a co-supervisor of PhD students from RWTH Aachen University, Ruhr-University Bochum (RUB) and Ludwig-Maximilians-University (LMU Munich). Since October 2018 C. Scheu is the mentor at the MPIE for female postdocs and group leaders to support their career in science, in a similar way as she has done during her time at the LMU where she was the mentor for the faculty Chemistry and Pharmacy.

The close connection to several universities is also demonstrated in joint projects funded by the German Science Foundation (DFG) via individual grants with A. Ludwig (Prof. at RUB) and L. Schmidt-Mende (Prof. at University of Konstanz) or within DFG Priority Programmes such as the SPP 1613 and SFB 761/3 "Steel ab initio". Research funding is also received from the Federal Ministry of Economic Affairs and Energy (BMWi) and the Leibniz Association (see p. 57). The research activities of some of these projects will be described in more detail below. The NG group is also very well integrated within the MPIE and strong collaborations have been established in the last years with the other MPIE departments and the Max Planck Fellow Group on Self-Reporting Materials. This is reflected in a large number of jointly supervised PhD students, collaborative research work, and a large number of joint publications in the field of correlative aberration-corrected STEM and atom probe investigations partly coupled with atomistic simulations [26-32], dewetting of Al thin films on Al_2O_3 [9,10], electrochemistry [1,6,7,33,34] and on Mo_2BC coatings [11-13,35,36] (see p. 183). Furthermore, the NG group has also an established network with international partners from Czech Republic, France, Israel, Sri Lanka and the USA.

C. Scheu is actively involved in the organization of national and international meetings and conferences. Recent examples are the co-organization of "Experimental and Theoretical insights on Interfaces of Ceramics" at the Conference on Electronic and Advanced Materials in Orlando, USA (2018) or "PS12 - Materials for Energy Production, Storage and Catalysis" at the 19th International Microscopy Congress (IMC19) in Sydney, Australia (2018). At the MPIE, she co-organized two workshops on "Challenges in Characterisation of Interfaces for Electrochemical Applications part I and part II" (2016/2017). In addition,

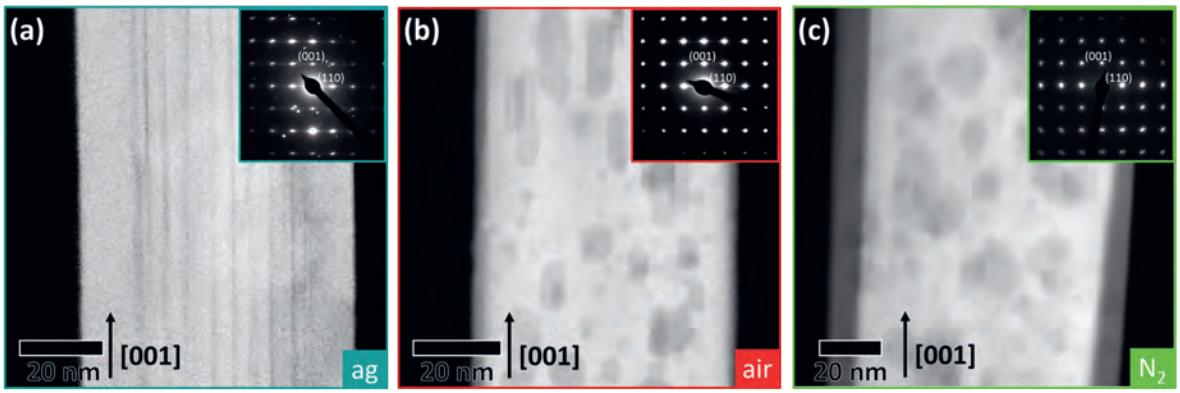


Fig. 2: *STEM images and corresponding diffraction patterns of rutile TiO_2 nanowires (a) as grown at 150°C, (b) after annealing at 500°C in air and (c) after annealing at 500°C in nitrogen. The nanowires show different defects depending on the heat treatment. Figure taken from [2].*

she organized the scientific colloquium dedicated to the 100 years anniversary of the MPIE (2017) and a meeting on "Hydrogen Technology" (2017). C. Scheu is also a co-organiser of the joint retreats "Mechanics meets Energy" between the department of Structure and Nano-/Micromechanics of Materials (SN) and the NG group.

The NG group is visible in the scientific community as shown by the number of invited and keynote talks at conferences and colloquia in universities and research institutions as follows. C. Scheu had keynote lectures at the EMRS Fall Meeting in Warsaw, Poland (2016) and at the MST conference in Salt Lake City, USA (2016). Her invited talks include the International Conference on Functional Nanomaterials and Nanodevices in Budapest, Hungary (2017), the 13th Multinational Congress on Microscopy, Rovinj, Croatia (2017), the Second Sino-German Symposium on Advanced Electron Microscopy and Spectroscopy of Materials, Xi'an China (2017) and the Grand Opening of UC Irvine Materials Research Institute, Irvine, USA (2018). All group members regularly attend national and international conferences and present their work in oral presentations or posters. Some notable achievements include S. Hieke receiving a travel award from the European Microscopy Society (EMS) to participate at the IMC19 in Sydney, Australia, and his nomination for the Young Scientist Assembly which was held prior to the IMC19 (2018), and L. Abdellaoui receiving the best oral communication award at the "1st International Meeting on Alternative and Green Energies" in Mohammedia, Morocco (2018).

Further awards to the NG group are the Alexander von Humboldt postdoc fellowship of J. Lim in the field of synthesis and advanced characterization of catalytic active, low-dimensional materials, and the nomination of C. Scheu to serve as a panel member for the strategic evaluation of the research field Matter of the Helmholtz Association. C. Scheu has also been elected and serves as a member for the evaluation board of the state North Rhine Westphalia "EFRE NRW 2014 – 2020 "NeueWerkstoffe. NRW".

The material portfolio studied within the NG group ranges from oxides [1-7,17-22], semiconductors [14-16,23], carbides [11-13] to metallic compounds [6-10] for applications such as photovoltaics, water splitting, fuel cells, conduction lines and functional coatings. Advanced characterisation methods, in particular, *ex situ* and *in situ* (S)TEM techniques are the link of the group members. In the following, the progress on three selected research examples in the field of materials for renewable energy applications is

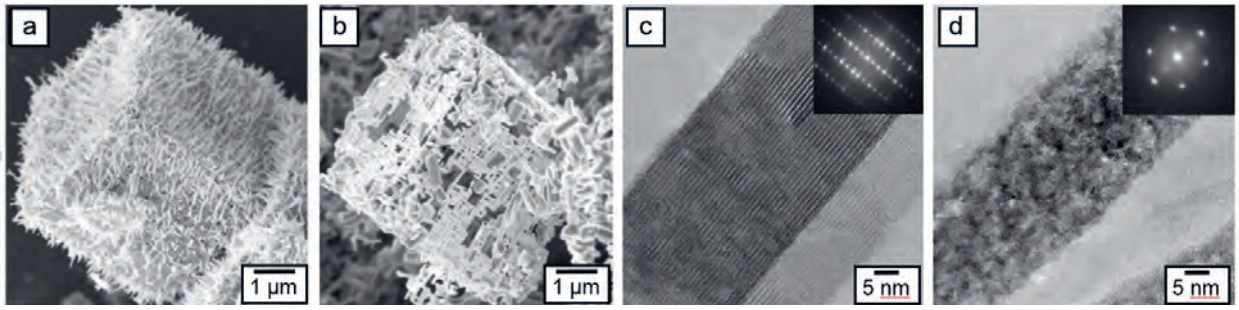


Fig. 3: *SEM images of (a) as grown $Nb_3O_7(OH)$ nanoarrays and (b) after ex situ annealing at 850°C. The ex situ annealing induced a phase transformation to H-Nb_2O_5. High resolution TEM images of an identical nanowire at (c) room temperature and (d) during in situ annealing at 800°C. Images and video frames taken from [5].*

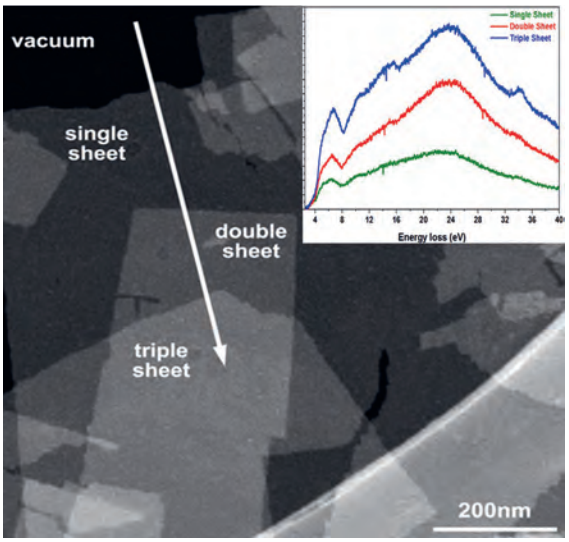


Fig. 4: *Low loss EELS data and STEM image of single, double and triple $Ca_2Nb_3O_{10}$ 2D nanosheets. Image reproduced from [40].*

described in detail, namely nanostructured transition metal oxides for photocatalytic application, materials for polymer-based fuel cells and thermoelectric materials for electricity generation.

Nanostructured Transition Metal Oxides for Photocatalytic Application

Transition metal oxides can be used as electrode material in energy storage devices, as support material in electrocatalysis or as photocatalyst. Large surface areas from nanostructuring introduce more reactive sites on their surface and shorter diffusion paths for charge carriers to reach the surface. The research activities, supported by the DFG, are based on TiO_2 and $Nb_3O_7(OH)$ nanostructures which are grown by NG members using facile hydrothermal-synthesis approaches.

The properties of TiO_2 nanoarrays were modified in the PhD thesis of A. Folger by using various heat treatments in different atmospheres [1-3]. These studies were done in close collaboration with Prof. L. Schmidt-Mende's group (University of Konstanz). Hydrothermally grown rutile TiO_2 nanoarrays possess a large number of defects (Fig. 2a) such as oxygen vacancies and tiny nanofingers on top, which act as recombination sites and limit the performance of functional devices [37]. Annealing in air eliminates these defects, but voids were formed in the interior of the nanowires [37]. As the involved mechanism remained open, *in situ* STEM heating experiments were performed which allowed to observe "live" the formation of the voids above 500 °C [1]. As shown by electron tomography and aberration-corrected STEM images, these voids are faceted and possess a Wulff-shape (Fig. 2b). They form via oxygen vacancy condensation. Moreover, above 250 °C, the hydroxide groups on the surface of the nanofingers are removed and a zipper-like condensation reaction closes the space between the nanofingers resulting in a single crystal [1].

The oxygen vacancy condensation has consequences on the electrical behaviour. In contrast to the as-grown nanowires, which are n-type semiconductors, the nanowires annealed in air are insulating [2]. By changing the annealing treatment from air to a slightly reducing N_2 atmosphere, the nanowires display a metal-like conductivity due to the formation of a 10 nm thin, sub-stoichiometric TiO_{2-x} shell (Fig. 2c). Within this shell, the oxidation state of Ti is mainly Ti^{3+} as found by energy loss near edge structure (ELNES) analysis, different from the Ti^{4+} state of the core. The rutile core still contains facetted voids [2].

An elegant way to introduce a different shell material on TiO_2 nanowires is the use of Si substrates and subsequent annealing treatments [3]. A 3-4 nm thin, amorphous SiO_x shell forms at 1050 °C, and homogenously covers the individual nanowires. In contrast to the nanowires heat-treated at lower temperatures, the core is free of lattice defects except for oxygen vacancies/Ti^{3+} ions at the area adjacent to the shell [3]. The shell is expected to increase the performance of photochemical devices and photovoltaic cells by minimizing carrier recombination. The presence of rutile seed layers on the morphology of hydrothermally grown rutile TiO_2 nanostructures was also studied [38]. Current activities include chemical etching of TiO_2 nanowires to increase the surface area further. Onto these hollow nanowires a Pt catalyst is deposited. The stability of these nanostructure is analyzed by J. Lim and R. Aymerich Armengol in close collaboration with O. Kasian and M. Ledendecker from the department of Interface Chemistry and Surface Engineering (GO).

Research activities on a blue TiO_2 photocatalyst deposited on an Al foil were initiated during a 3-month visit of J. Bandara (Prof. at the National Institute of Fundamental Studies, Sri Lanka) who was granted a renewed research stay from the Alexander von Humboldt Foundation. The joint research unravelled the mechanism responsible for the observed superior hydrogen production during UV illumination [39]. Detailed microstructural characterization of A. Folger and S. Zhang showed that Ti^{3+} and Ti^{2+} states are present at the surface of TiO_2 nanoparticles. They are stabilized by an aluminium hydroxide oxide layer surrounding them. These reduced states are continuously self-generated via absorption of UV light and deliver the electrons required for the hydrogen generation [39].

The functional properties of a $Nb_3O_7(OH)$ photocatalyst and its modifications by doping and phase transformation were investigated by S. Betzler (now at: Lawrence Berkeley National Laboratory, USA) in her PhD thesis [4,5,40] together with B. Lotsch (Di-

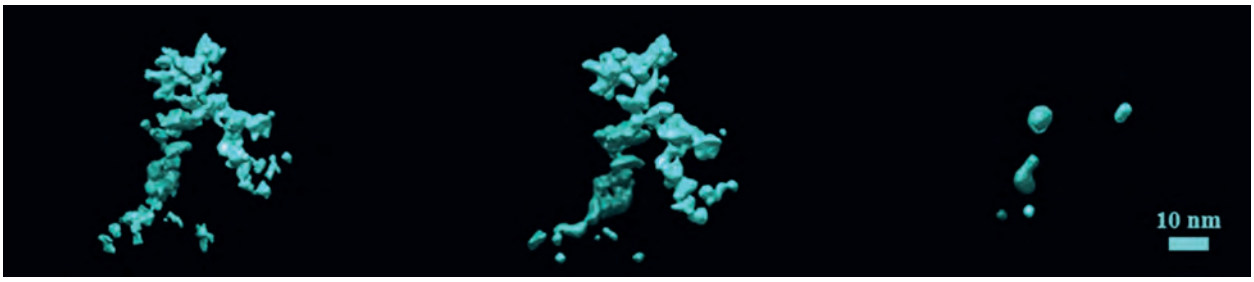


Fig. 5: *Electron tomography reconstructions of an identical Pt/Ru catalyst network before (left) and after 2000 cycles (middle) and 7000 cycles (right) of a cyclic voltammetry experiment performed at [0-1.2]V_{RHE} in saturated $HClO_4$. Strong dissolution is observed. Modified image from [8].*

rector at the MPI for Solid State Research, Stuttgart) and L. Schmidt-Mende. 3D nanoarrays consisting of nanowires with a width of few tens of nanometres and a length of micrometres which are arranged perpendicular to each other can be grown with the developed hydrothermal synthesis strategy [40]. Incorporation of Ti within the orthorhombic crystal structure leads to a higher hydrogen production rate in UV light [4]. Up to 12 at.% Ti can be homogeneously incorporated in the crystal lattice as proven by EDX and EELS measurements in the STEM. Ti not only lowers the number of surface defects at the nanowires, but also leads to changes in the aspect ratio resulting in a morphology with a larger surface area [4]. The $Nb_3O_7(OH)$ morphology can be also modified by the type of precursor used as observed in the master thesis of T. Gänsler. He will continue working in this field during his PhD at the MPIE. In general, other factors such as temperature and time are also important for the growth of nanostructures via hydro/solvothermal synthesis strategies as demonstrated by A. Frank in her PhD thesis [14-16].

A different approach to modify the functional properties of the $Nb_3O_7(OH)$ nanoarrays is the thermally induced phase transformation to Nb_2O_5 [5]. The structural changes of the 3D networks were investigated both *ex situ* in air and *in situ* under reducing atmosphere in the TEM (Fig. 3). The *in situ* TEM studies reveal the release of water from the crystal structure and the formation of an oxygen-deficient niobium oxide phase. Thereby, stacking faults play an important role, which are mainly located at the interior of the nanowires. At these defective regions, dislocations form at ~450 °C, which move towards the surface. This initiates the phase transformation which is completed at temperatures >750 °C. To compensate for the volume differences of the original $Nb_3O_7(OH)$ crystal and the oxygen-deficient niobium oxide, pores are formed (Fig. 3) [5]. The *in situ* TEM also reveals an extremely high thermal stability of the nanowire network, as confirmed by *ex situ* heating studies. The 3D morphology remains up to 850°C where merging of nanowires occurs (Fig. 3). Due to the constant supply of oxygen, the *ex situ* annealing in air leads to a different niobium oxide phase. Stoichiometric Nb_2O_5 possessing a monoclinic crystal structure is formed [5]. The *in situ* TEM studies were performed in close collaboration with T. Harzer and G. Dehm from the Department of Structure and Nano-/Micromechanics of Materials (SN).

The differences in electronic structure and properties between $Nb_3O_7(OH)$ and H-Nb_2O_5 have been evaluated by comparing experimental data with density functional theory (DFT) calculations performed by the group of J. Minar (Prof. University of West Bohemia, Plzen, Czech Republic) [41]. The band gap calculated by the modified Becke-Johnson (mBJ) potential is in good agreement with both UV-VIS measurements and low loss EELS. Also the calculated low loss EELS region and O-K, and Nb-$M_{2,3}$ ELNES agree well with the experiment. The DFT calculations reveal that both compounds are n-type semiconductors with a similar electron mobility but higher hole mobility of $Nb_3O_7(OH)$ [41].

Further promising UV active photocatalysts are 2D nanosheets based on niobates obtained via exfoliation of different perovskite parent phases [19,42,43]. These studies are done in collaboration with the group of B. Lotsch who synthesized the material and explored the functional properties while the NG contributes to atomic scale characterization (Fig. 4). Monochromated EELS measurements were used to determine the band gap of 2D $Ca_2Nb_3O_{10}$ nanosheets with a high spatial resolution of <10 nm [19]. In contrast to most nanostructures where the band gap increases due to quantum size effect, the value of the $Ca_2Nb_3O_{10}$ nanosheets was similar to that of the bulk parent phase $KCa_2Nb_3O_{10}$. This unexpected result can be explained with the help of DFT calculations, which show that the partial density of states related to the K atoms is not affecting the band gap value. Currently, S. Zhang performs *in situ* heating experiments to understand the growth mechanism of NiO_x nanoparticles on the 2D $Ca_2Nb_3O_{10}$ nanosheets. These sheets are also interesting for lighting application, when doped with lanthanides. R. Changizi investigated in her master thesis successfully the doped bulk parent phase with cathodoluminescence experiments and she will continue to conduct such measurements at the nanosheets in her PhD thesis.

Exploring materials for solar hydrogen production is also conducted by the NG group within the DFG priority programme 1613 “Fuels Produced

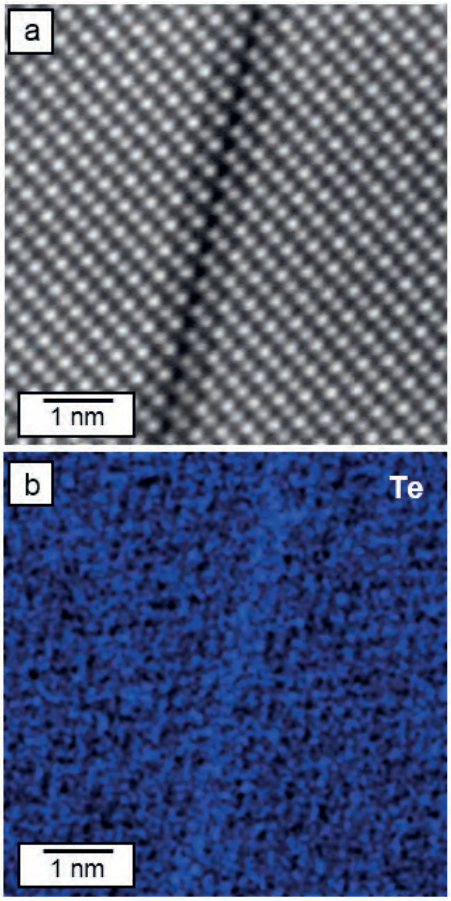


Fig. 6: *STEM image and corresponding tellurium map of an as-quenched $AgSbTe_2$ thermoelectric material. The figure is adapted from [50].*

Regeneratively Through Light-Driven Water Splitting: Clarification of the Elemental Processes Involved and Prospects for Implementation in Technological Concepts". After a positive evaluation, the NG group continued in the second funding period to carry out scale bridging microstructural characterisation of novel materials synthesized by project partners within the SPP 1613/2. The activities of A. Müller during his PhD thesis [17,18] and S. Zhang in collaboration with the groups of T. Bein (Prof. at LMU Munich), D. Fattakhova-Rohlfing (Prof. at FZ Jülich), R. Pentcheva (Prof. at University of Duisburg-Essen), A. Ludwig and W. Schuhmann (both Prof. at RUB) and A. Fischer (Prof. at University of Freiburg) have led to several joint publications in the field of photoabsorbers (Sn-doped Fe_2O_3 [21,20], Fe_2O_3/WO_3 [17]), photoanodes (zinc ferrite [44], Sn doped Fe_2O_3 [20], Mo-doped $BiVO_4$ [45]), photocathodes (FeCrAl oxide [46]) and various catalyst [47,48]. Only new publications since the last scientific report 2013-2015 are included.

In line with this activities, the NG group studied together with T. Li (Jun.-Prof. RUB), O. Kasian and K. Mayrhofer (both GO), B. Gault and D. Raabe (both department of Microstructure Physics and Alloy Design (MA)) Iridium-oxide based catalyst for the oxygen evolution reaction (OER) for water electrolysis [34] (see p. 163). Another interesting approach is the use of multinary noble metal free catalyst which are explored together with A. Ludwig and W. Schuhmann [49]. They are fabricated by co-sputtering several elements into an ionic liquid and A. Garzón-Manjón performs *ex situ* and *in situ* STEM experiments to unravel their crystal structure and phase transformation [50].

Materials for Polymer-based Fuel Cells

The hydrogen produced by solar-driven water splitting or water electrolysis can be used in fuel cells to generate electricity for households or driving vehicles without the release of harmful by-products during operation. C. Scheu and her team members are working on polymer-based fuel cells since 2010, together with industry partners. The centrepiece of the polymer-based fuel cells is the membrane electrode assembly with the electrode consisting of a high surface area support material on which a catalyst is deposited. This assembly is prone to degradation and the aim of the studies is to discover the underlying mechanism. The goal is to develop guidelines for material selection with improved lifetime and reliability. For example, C. Scheu and her team have successfully determined how the polymer membrane type and molecular weight distribution of the polymer chains affect the degradation leading to poisoning of the cathode layer [24]. A narrow molecular weight distribution is optimal as long chains block the catalyst surface while short chains are able to move very far into the microporous layer hindering efficient mass transport. They also discovered that cross-linkers and the addition of inorganic particles can be successfully used to minimize degradation [24].

The electrode materials are also affected by operation related degradation effects. In the PhD thesis of K. Hengge [6-8], the commonly used Pt catalyst in the anode was replaced by a Pt/Ru catalyst and its behaviour during various fuel cell operating conditions was analysed in detail [7,8]. These studies were done in close collaboration with the group of K. Mayrhofer (GO). Cyclic voltammetry and STEM experiments were performed on identical catalyst nanoparticles located on their carbon support. The change in chemical composition was studied with EDX and electron tomography was used to monitor the surface and volume changes of the individual nanoparticles (Fig. 5). Dissolution and agglomeration are the dominant degradation mechanisms occurring in the dynamic fuel cell operation mode [7]. Both depend strongly on the applied voltage with dissolution and agglomeration increasing when the upper potential increased. The catalyst dissolution has a strong effect on the real stack performance and the formation of a catalyst band within the polymer membrane after continuous fuel cell operation was observed [8]. The position of the band depends on the ratio of the molar flux of H_2 and O_2.

In an earlier work, the carbon support had been replaced by non-stoichiometric tungsten oxide WO_{3-x} where novel platinum networks grow instead of nanoparticles [51]. The assembled fuel cells possessed a long-term stability related to the stability of the Pt networks [51], however, its growth mechanism remained unknown. K. Hengge was able to close this knowledge gap in her PhD by performing systematic studies to understand the formation process [6]. In the first step of the synthesis, the liquid precursor is deposited on the WO_{3-x} and is thermally reduced in the second step. The reduction begins at the surface of the initially formed Pt and Cl^--containing truncated bulk octahedral solid [6]. It progresses with time into the interior which consists of an amorphous matrix in which Pt nanocrystals are embedded while the exterior is already the network of interconnected Pt nanocrystals. After a reduction time of only 20 minutes, the Cl^- is completely removed and a Pt network with pores of different sizes remains. With a further increase in the reaction time, networks with a higher surface area can be produced [6].

As degradation is a major problem not only in fuel cells but also battery materials, similar experiments are planned for novel electrode materials. First experiments are done by R. Sahu, K. Hengge and J. Lim on a copper hexacyanoferrate electrode prepared by F. La Mantia (Prof. at Bremen University) in cooperation with K. Schweinar and D. Raabe (MA).

Thermoelectric Materials for Electricity Generation

Thermoelectric materials can be used to generate electricity from waste heat through the Seebeck effect. The conversion efficiency can be increased by introducing defects that efficiently scatter phonons, but not electrons to keep a good electrical conductivity. In the ongoing PhD thesis of L. Abdellaoui and the bachelor thesis of R. Bueno Villoro several promising thermoelectric materials are explored together with the project partners O. Cojocaru-Mirédin (RWTH Aachen), Y. Amouyal (Prof. at Technion, Israel), and the MA department [29-31].

In particular, electron channeling contrast imaging in a scanning electron microscope is used together with S. Zafferer (MA) to analyse the density of stacking faults in a quantitative manner. Stacking faults in as-quenched $AgSbTe_2$ act as scattering sites for phonons leading to a strong reduction of the lattice thermal conductivity. A much higher density of stacking faults is observed at small-angle grain boundaries. They are further characterized at the atomic scale using Z-contrast imaging in STEM and EDX mapping (Fig. 6). These results are complemented with 3D atom probe tomography studies of the groups of O. Cojocaru-Mirédin and B. Gault (MA) to determine the chemistry of the stacking faults with high accuracy. The results show that Ag is depleted, and Sb/Te are enriched at the stacking faults [52]. The phase stability of the $AgSbTe_2$ compound will be investigated by *ex situ* and *in situ* annealing experiments. Such studies have been very successfully conducted in the NG group in the PhD theses of S. Gleich on Mo_2BC coatings [13] and by S. Hieke on thin Al films on Al_2O_3 substrates [9,10] (see p.183).

References

1. Folger, A.; Ebbinghaus, P.; Erbe, A.; Scheu, C.: ACS Appl Mater Inter 9 (2017) 13471.
2. Folger, A.; Kalb, J.; Schmidt-Mende, L.; Scheu, C.: Nanomaterials S-Basel 7 (2017) 289.
3. Folger, A.; Kalb, J.; Schmidt-Mende, L.; Scheu, C.: Apl Mater 5 (2017) 086101.
4. Betzler, S.B.; Podjaski, F.; Beetz, M.; Handloser, K.; Wisnet, A.; Handloser, M.; Hartschuh, A.; Lotsch, B.V.; Scheu, C.: Chem Mater 28 (2016) 7666.
5. Betzler, S.B.; Harzer, T.; Ciston, J.; Dahmen, U.; Dehm, G.; Scheu, C.: Cryst Growth Des 16 (2016) 4309.
6. Hengge, K.A.; Heinzl, C.; Perchthaler, M.; Geiger, S.; Mayrhofer, K.J.J.; Scheu, C.: Cryst Growth Des 17 (2017) 1661.
7. Hengge, K.; Gansler, T.; Pizzutilo, E.; Heinzl, C.; Beetz, M.; Mayrhofer, K.J.J.; Scheu, C.: Int J Hydrogen Energ 42 (2017) 25359.
8. Hengge, K.; Heinzl, C.; Perchthaler, M.; Varley, D.; Lochner, T.; Scheu, C.: J Power Sources 364 (2017) 437.
9. Hieke, S.W.; Breitbach, B.; Dehm, G.; Scheu, C.: Acta Mater 133 (2017) 356.
10. Hieke, S.W.; Dehm, G.; Scheu, C.: Acta Mater 140 (2017) 355.
11. Gleich, S.; Fager, H.; Bolvardi, H.; Achenbach, J.O.; Soler, R.; Pradeep, K.G.; Schneider, J.M.; Dehm, G.; Scheu, C.: J Appl Phys 122 (2017) 0753051.
12. Gleich, S.; Soler, R.; Fager, H.; Bolvardi, H.; Achenbach, J.O.; Hans, M.; Primetzhofer, D.; Schneider, J.M.; Dehm, G.; Scheu, C.: Mater Design 142 (2018) 203.
13. Gleich, S.; Breitbach, B.; Peter, N.J.; Soler, R.; Bolvardi, H.; Schneider, J.M.; Dehm, G.; Scheu, C.: Surf Coat Tech 349 (2018) 378.
14. Frank, A.; Wochnik, A.S.; Bein, T.; Scheu, C.: Rsc Advances 7 (2017) 20219.
15. Frank, A.; Grunwald, J.; Breitbach, B.; Scheu, C.: Nanomaterials S-Basel 8 (2018) 405.
16. Frank, A.; Changizi, R.; Scheu, C.: Micron 109 (2018) 1.
17. Müller, A.; Kondofersky, I.; Folger, A.; Fattakhova-Rohlfing, D.; Bein, T.; Scheu, C.: Mater Res Express 4 (2017) 016409.

18. Müller, A.; Peglow, S.; Karnahl, M.; Kruth, A.; Junge, H.; Brüser, V.; Scheu, C.: Nanomaterials S-Basel 8 (2018) 502.
19. Virdi, K.S.; Kauffmann, Y.; Ziegler, C.; Ganter, P.; Blaha, P.; Lotsch, B.V.; Kaplan, W.D.; Scheu, C.: J Phys Chem-US C 120 (2016) 11170.
20. Zhang, S.Y.; Scheu, C.: Microscopy 67 (2018) i133.
21. Hufnagel, A.G.; Hajiyani, H.; Zhang, S.; Li, T.; Kasian, O.; Gault, B.; Breitbach, B.; Bein, T.; Fattakhova-Rohlfing, D.; Scheu, C.; Pentcheva, R.: Adv Funct Mater (2018) 1804472 .
22. Dennenwaldt, T.; Wisnet, A.; Sedlmaier, S.J.; Doblinger, M.; Schnick, W.; Scheu, C.: Micron 90 (2016) 6.
23. Venkatesan, S.; Mancabelli, T.; Krogstrup, P.; Hartschuh, A.; Dehm, G.; Scheu, C.: J Appl Phys 121 (2017) 085702.
24. Ossiander, T.; Perchthaler, M.; Heinzl, C.; Schonberger, F.; Volk, P.; Welsch, M.; Chromik, A.; Hacker, V.; Scheu, C.: J Membrane Sci 509 (2016) 27.
25. Wagatha, P.; Pust, P.; Weiler, V.; Wochnik, A.S.; Schmidt, P.J.; Scheu, C.; Schnick, W.: Chem Mater 28 (2016) 1220.
26. Liebscher, C.H.; Yao, M.; Dey, P.; Lipinska-Chwalek, M.; Berkels, B.; Gault, B.; Hickel, T.; Herbig, M.; Mayer, J.; Neugebauer, J.; Raabe, D.; Dehm, G.; Scheu, C.: Phys Rev Mat 2 (2018) 023804.
27. Liebscher, C.H.; Stoffers, A.; Alam, M.; Lymperakis, L.; Cojocaru-Miredin, O.; Gault, B.; Neugebauer, J.; Dehm, G.; Scheu, C.; Raabe, D.: Phys Rev Lett 121 (2018) 015702.
28. Stoffers, A.; Barthel, J.; Liebscher, C.H.; Gault, B.; Cojocaru-Miredin, O.; Scheu, C.; Raabe, D.: Microsc Microanal 23 (2017) 291.
29. Cojocaru-Miredin, O.; Abdellaoui, L.; Nagli, M.; Zhang, S.Y.; Yu, Y.; Scheu, C.; Raabe, D.; Wuttig, M.; Amouyal, Y.: ACS Appl Mater Inter 9 (2017) 14779.
30. Yu, Y.; He, D.S.; Zhang, S.Y.; Cojocaru-Miredin, O.; Schwarz, T.; Stoffers, A.; Wang, X.Y.; Zheng, S.Q.; Zhu, B.; Scheu, C.; Wu, D.; He, J.Q.; Wuttig, M.; Huang, Z.Y.; Zu, F.Q.: Nano Energy 37 (2017) 203.
31. Yu, Y.; Zhang, S.Y.; Mio, A.M.; Gault, B.; Sheskin, A.; Scheu, C.; Raabe, D.; Zu, F.Q.; Wuttig, M.; Amouyal, Y.; Cojocaru-Miredin, O.: ACS Appl Mater Inter 10 (2018) 3609.
32. Yao, M.J.; Welsch, E.; Ponge, D.; Haghighat, S.M.H.; Sandlobes, S.; Choi, P.; Herbig, M.; Bleskov, I.; Hickel, T.; Lipinska-Chwalek, M.; Shanthraj, P.; Scheu, C.; Zaefferer, S.; Gault, B.; Raabe, D.: Acta Mater 140 (2017) 258.
33. Toparli, C.; Hieke, S.W.; Altin, A.; Kasian, O.; Scheu, C.; Erbe, A.: J Electrochem Soc 164 (2017) II734.
34. Li, T.; Kasian, O.; Cherevko, S.; Zhang, S.; Geiger, S.; Scheu, C.; Felfer, P.; Raabe, D.; Gault, B.; Mayrhofer, K.J.J.: Nat Catal 1 (2018) 300.
35. Soler, R.; Gleich, S.; Kirchlechner, C.; Scheu, C.; Schneider, J.M.; Dehm, G.: Mater Design 154 (2018) 20.
36. Djaziri, S.; Gleich, S.; Bolvardi, H.; Kirchlechner, C.; Hans, M.; Scheu, C.; Schneider, J.M.; Dehm, G.: Surf Coat Tech 289 (2016) 213.
37. Wisnet, A.; Bader, K.; Betzler, S.B.; Handloser, M.; Ehrenreich, P.; Pfadler, T.; Weickert, J.; Hartschuh, A.; Schmidt-Mende, L.; Scheu, C.; Dorman, J.A.: Adv Funct Mater 25 (2015) 2601.
38. Kalb, J.; Dorman, J.A.; Folger, A.; Gerigk, M.; Knittel, V.; Pluisch, C.S.; Trepka, B.; Lehr, D.; Chua, E.; Goodge, B.H.; Wittemann, A.; Scheu, C.; Polarz, S.; Schmidt-Mende, L.: J Cryst Growth 494 (2018) 26.
39. De Silva, N.L.; Jayasundera, A.C.A.; Folger, A.; Kasian, O.; Zhang, S.; Yan, C.-F.; Scheu, C.; Bandara, J.: Catal Sci Technol 8 (2018) 4657.
40. Betzler, S.B.; Wisnet, A.; Breitbach, B.; Mitterbauer, C.; Weickert, J.; Schmidt-Mende, L.; Scheu, C.: J Mater Chem A 2 (2014) 12005.
41. Khan, W.; Betzler, S.B.; Sipr, O.; Ciston, J.; Blaha, P.; Scheu, C.; Minar, J.: J Phys Chem-US C 120 (2016) 23329.
42. Ziegler, C.; Dennenwaldt, T.; Weber, D.; Duppel, V.; Kamella, C.; Podjaski, F.; Tuffy, B.; Moudrakovski, I.; Scheu, C.; Lotsch, B.V.: Z Anorg Allg Chem 643 (2017) 1668.
43. Ganter, P.; Ziegler, C.; Friedrichs, A.T.; Duppel, V.; Scheu, C.; Lotsch, B.V.: ChemNanoMat 3 (2017) 411.
44. Hufnagel, A.G.; Peters, K.; Muller, A.; Scheu, C.; Ng, D.F.R.; Bein, T.: Adv Funct Mater 26 (2016) 4435.
45. Rohloff, M.; Anke, B.; Zhang, S.Y.; Gernert, U.; Scheu, C.; Lerch, M.; Fischer, A.: Sustainable Energy Fuels 1 (2017) 1830.
46. Kondofersky, I.; Muller, A.; Dunn, H.K.; Ivanova, A.; Stefanic, G.; Ehrensperger, M.; Scheu, C.; Parkinson, B.A.; Fattakhova-Rohlfing, D.; Bein, T.: J Am Chem Soc 138 (2016) 1860.
47. Fominykh, K.; Chernev, P.; Zaharieva, I.; Sicklinger, J.; Stefanic, G.; Doblinger, M.; Muller, A.; Pokharel, A.; Bocklein, S.; Scheu, C.; Bein, T.; Fattakhova-Rohlfing, D.: ACS Nano 9 (2015) 5180.
48. Fominykh, K.; Böhm, D.; Zhang, S.Y.; Folger, A.; Döblinger, M.; Bein, T.; Scheu, C.; Fattakhova-Rohlfing, D.: Chem Mater 29 (2017) 7223.
49. Löffler, T.; Meyer, H.; Savan, A.; Wilde, P.; Garzón Manjón, A.; Ting Chen, Y.T.; Ventosa, E.; Scheu, C.; Ludwig, A.; Schuhmann, W.: Adv Energy Mater (2018) 1802269.
50. Garzón-Manjón, A.; Meyer, H.; Grochla, D.; Löffler, T.; Schuhmann, W.; Ludwig, A.; Scheu, C.: Nanomaterials S-Basel 8 (2018) 903.
51. Heinzl, C.; Hengge, K.A.; Perchthaler, M.; Hacker, V.; Scheu, C.: J Electrochem Soc 162 (2015) F280.
52. Abdellaoui, L.; Zhang, S.; Zaefferer, S.; Cojocaru-Miredin, O.; Baranovskiy, A.; Amouyal, Y.; Raabe, D.; Scheu, C.: To be submitted (2018).

Research Projects in Progress

Abdellaoui, Bueno Villoro, Scheu (with S. Zaefferer, B. Gault*, D. Raabe*, O. Cojocaru-Miredin**, Y. Amouyal*** (*MA Department, **RWTH Aachen, ***Technion, Israel))*: Microstructural and spectroscopic analysis of thermoelectrics

*Changizi, Scheu (with T. Schwarz, *MA Department):* Combining cathodoluminescence studies of functional materials with structural investigations

Frank, Scheu (*with A. Kruth*, V. Presser**, *Leibniz Institute for Plasma Science and Technology, Greifswald, ** Leibniz Institute for New Materials in Saarbrücken):* Characterisation of nanohybrids for renewable energy applications *(Leibniz Project)*

Gänsler, Scheu (with S. Betzler, B. Lotsch**, L. Schmidt-Mende*** (*Lawrence Berkeley National Laboratory, USA, **LMU Munich and MPI for Solid State Research, ***University of Konstanz))*: Tailoring functional properties of niobium hydroxide *(DFG Project)*

Garzón-Manjón, Scheu (with A. Ludwig, RUB University): *Ex situ* and *in situ* studies of binary and multinary nanoparticles grown in ionic liquids *(DFG Project)*

Hieke, Scheu (with G. Dehm, SN Department): Stability of thin films under harsh environments

Lim, Aymerich Armengol, Scheu (with O. Kasian, M. Ledendecker, GO Department): Synthesis and characterization of catalytic active, low-dimensional materials (J. Lim supported by AvH)

Hengge, Obermaier, Scheu: Investigation of degradation mechanism in polymer based fuel cells

Sahu, Scheu (with J. Schneider (*RWTH Aachen))*: Characterization of nanolaminated and 2D materials by electron microscopy and spectroscopy

Scheu (with M. Lipinska-Chwalek and J. Mayer, RWTH Aachen)): Microstructure characterization – local structure, defect and chemical analysis (SFB 761/3 partial project C01)

Zhang, Scheu (with T. Bein, D. Fattakhova-Rohlfing***, R. Pentcheva** (*LMU Munich, **Forschungszentrum Jülich, ***University of Duisburg)*: Investigation of photo-electrochemical water splitting-active nanostructures

Max Planck Fellow Group on Self-Reporting Materials

Group Head: J. M. Schneider

MATERIALS CHEMISTRY | RWTH AACHEN UNIVERSITY

A: Self-Reporting Materials:

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. conceivable 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.

One material property that can be measured in service is the electrical resistivity which is a measure for how strongly a material opposes the flow of an electric current. The measurement is non-destructive. Within the Max Planck Fellow Group the resistivity changes of magnetron sputtered, amorphous Cr_2AlC thin films were measured during heating in vacuum for the first time. For this Pt electrodes were deposited onto the as deposited Cr_2AlC thin films. Our previous work has established that annealing amorphous Cr_2AlC triggers the transformation into a disordered, hexagonal solid solution phase [1]. Subsequently, upon further heating the disordered solid solution phase transforms into the (ordered) hexagonal MAX phase [1]. Hence, amorphous Cr_2AlC was selected for this proof of concept study.

Fig. 1 shows the results of in situ resistivity measurements during heating as well as the structural changes of samples (monitored by ex situ X-ray diffraction (XRD)) and previously published differential scanning calorimetry (DSC) data [1]. In Fig. 2 bright field images and selected area electron diffraction (SAED) patterns of focused ion beam (FIB) lamellas extracted from Cr_2AlC coatings after annealing to temperatures from 500 to 800 °C in vacuum are shown.

Based on the correlative XRD data from Fig.1, the SAED from Fig.2, and the DSC data from Fig.1 it is evident that the resistivity changes at approx. 552 °C indicate the phase transitions from amorphous to a disordered hexagonal solid solution and that the resistivity changes at approx. 585 °C signal the phase transition from disordered hexagonal solid solution to the ordered MAX phase. Hence, we have shown that

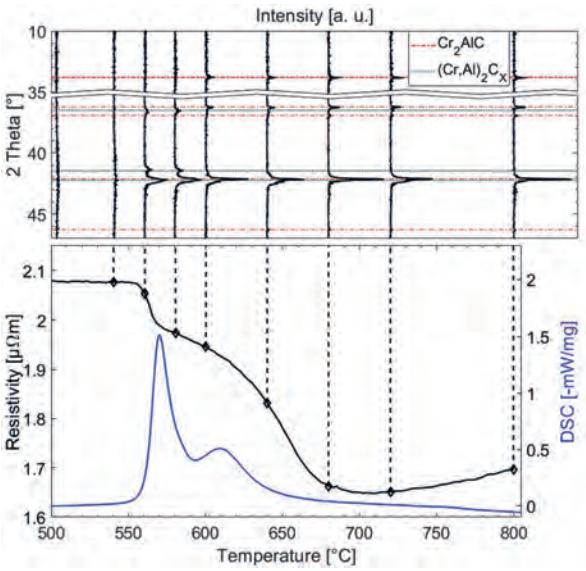


Fig. 1: *X-ray diffraction data of Cr_2AlC samples annealed to various temperatures up to 800 °C and resistivity measured while annealing (black) [unpublished] compared to DSC results (blue) [1].*

thermally stimulated phase changes in Cr_2AlC thin films can be revealed by in situ resistivity measurements. These results underline the potential of in situ resistivity measurements for monitoring structural change in Cr_2AlC coatings during application.

B. Other research topics in collaboration with the MPIE:

B1. Quantum mechanically guided design of ultra strong and damage tolerant glasses (In collaboration with G. Dehm & D. Raabe & B. Gault)

The enhancement of fracture toughness, see Fig. 3, and strength of a cobalt tantalum-based metallic glass thin film with increasing boron content was recently reported [2].

The improvement of the mechanical performance is attributed to the formation of a compositionally lamel-

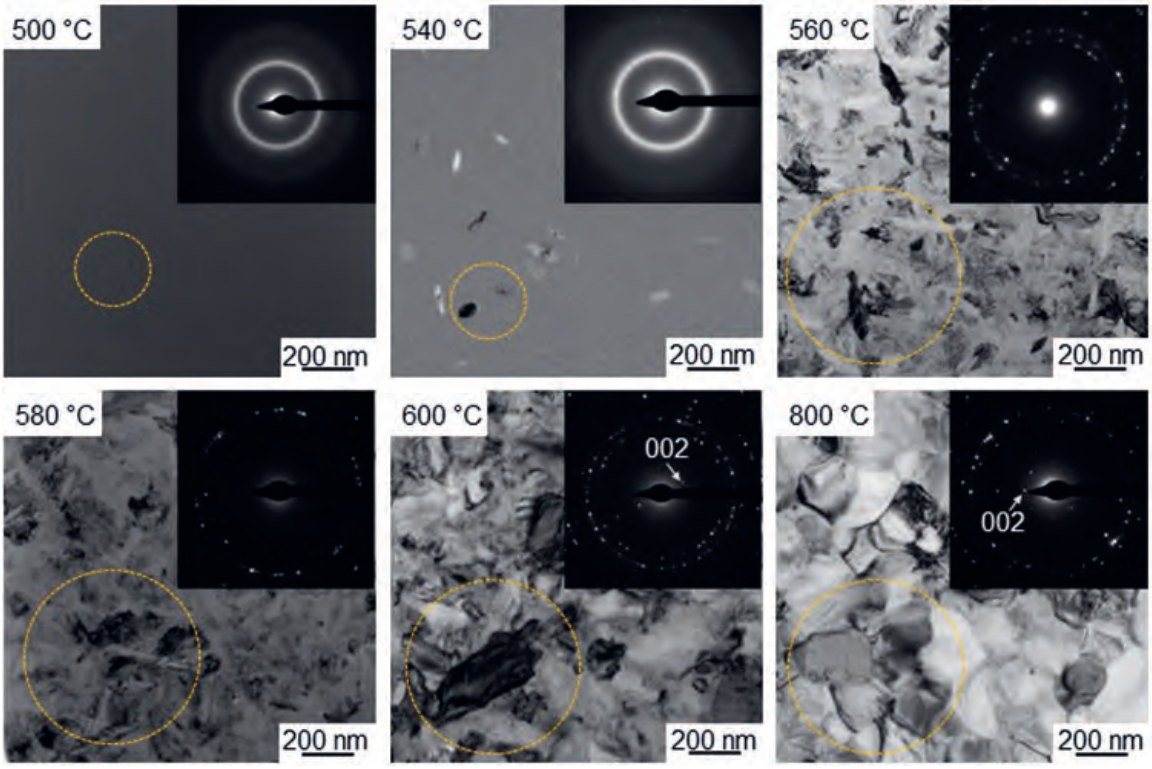


Fig. 2: *Bright field images and SAED patterns of FIB lamellas extracted from Cr_2AlC coatings after annealing at 500, 540, 560, 580, 600, and 800 °C in vacuum [unpublished].*

lar compared to uniform glass microstructure, which becomes thinner with increasing boron content as revealed by transmission electron microscopy, see Fig. 4. Compositional variations across the lamellar structure are revealed by atom probe tomography (APT), see Fig. 4.

These findings provide a new design route for cobalt tantalum-based metallic glass thin films with enhanced mechanical performance as a function of boron content. A compositional lamellar structure revealed by TEM shows that the lamella thickness decreases as boron content increases. A substantial compositional difference was observed within the lamellar structure, where boron- and cobalt-rich structures were revealed by APT. As the thickness of the lamellar structure decreases, fracture toughness

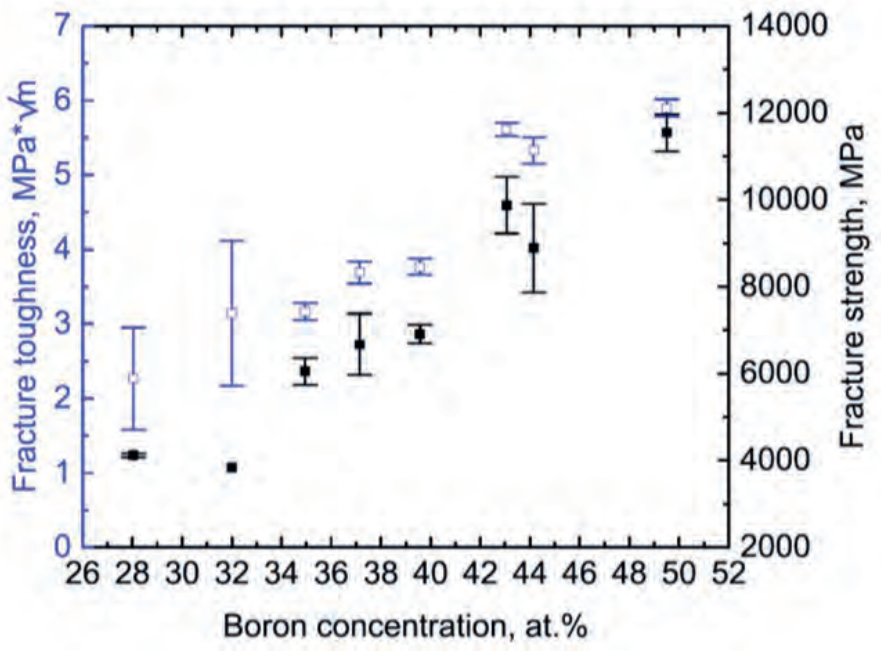


Fig. 3: *Micro-mechanical testing of the combinatorial CoTaB metallic glass thin film. Fracture toughness and fracture strength graphs as a function of boron content as revealed by microcantilever bending tests [2].*

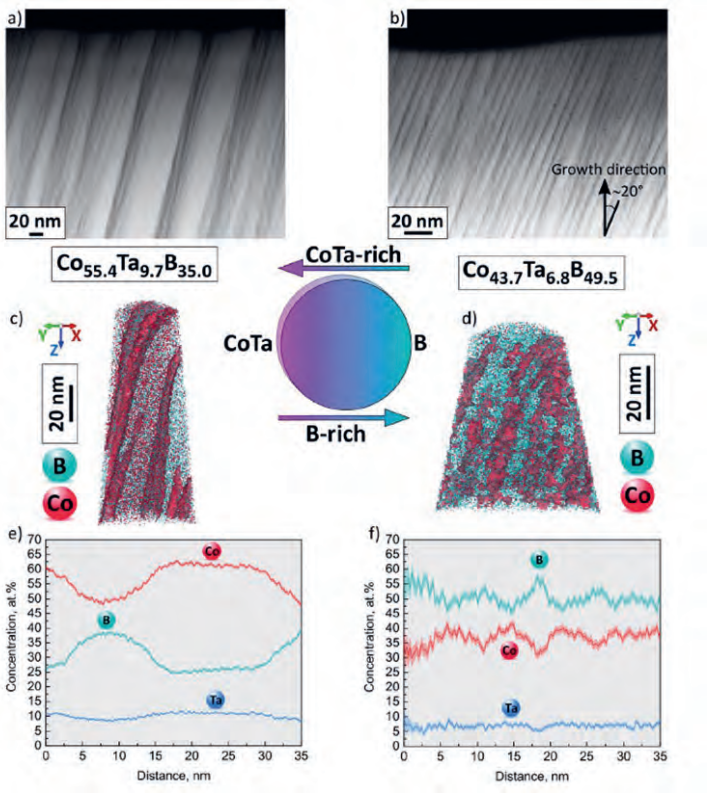


Fig. 4: *a, b) HAADF-STEM micrographs revealing the lamellar structure for $Co_{55.4}Ta_{9.7}B_{35.0}$ and $Co_{43.7}Ta_{6.8}B_{49.5}$. c, d) Corresponding APT reconstructions from $Co_{55.4}Ta_{9.7}B_{35.0}$ and $Co_{43.7}Ta_{6.8}B_{49.5}$ compositions. e, f) Compositional profiles perpendicular to lamellar structures revealing the boron and cobalt variations [2].*

and fracture strength are enhanced, suggesting the origin of the enhanced mechanical properties in the nanolaminated structure. The origin of the lamella formation is subject of ongoing research.

B2. Can high strength and moderate ductility be combined in wear resistant coatings?

A fundamental plasticity study of X_2BC nanolaminates (X=Hf, Mo) in collaboration with C. Scheu & G. Dehm [3-6].

These activities are described on p. 183.

References

1. Abdulkadhim, A.; to Baben, M.; Takahashi, T.; Schnabel, V.; Hans, M.; Polzer, C.; Polcik, P.; Schneider, J. M.: Surf Coat Technol 206 (2011) 599.
2. Kontis, P.; Köhler, M.; Evertz, S.; Chen, Y.-T.; Schnabel, V.; Soler, R.; Bednarick, J.; Kirchlechner, C.; Dehm, G.; Raabe, D.; Schneider, J. M.; Gault, B.: Scripta Mater 155 (2018) 73.
3. Djaziri, S.; Gleich, S.; Bolvardi, H.; Kirchlechner, C.; Hans, M.; Scheu, C.; Schneider, J.M.; Dehm; G.: Surf Coat Technol 289 (2016) 213.
4. Gleich, S.; Fager, H.; Bolvardi, H.; Achenbach, J. -O.; Soler, R.; Pradeep, K. G.; Schneider, J. M.; Dehm, G.; Scheu; C.: J Appl Phys 122 (2017) 075305.
5. Soler, R.; Gleich, S.; Kirchlechner, C.; Scheu, C.; Schneider, J.M.; Dehm, G.: Mater Des 154 (2018) 20.
6. Gleich, S.; Breitbach, B.; Peter, N.J.; R. Soler, H. Bolvardi, J.M. Schneider, G. Dehm, C. Scheu: Surf Coat Technol 349 (2018) 378.

Max Planck Research Group on High Temperature Materials

G. Eggeler [1], A. Kostka [2], X. Wu [3,4,5], L. Cao [1,6]

[1] Chair of Material Science and Engineering, Ruhr-Universität Bochum
[2] now at: Center for Interface-Dominated High Performance Materials (ZGH), Ruhr-Universität Bochum
[3] Department of Microstructure Physics and Alloy Design (MA)

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 HTM group. The expertise of the group lies in the field of exploring and explaining microstructural evolution during processing and service of engineering materials. Research of the group in the reporting period has shifted from tempered martensite ferritic steels (last reporting period) to single crystal Ni-base superalloys. Gunther Eggeler is the speaker of the collaborative research center SFB/TR 103 (From Atoms to Turbine Blades – A Scientific Basis for a New Generation of Single Crystal Superalloys), which interacts closely with the TEM and atom probe tomography researchers at the MPIE. Research in the group is focused on elementary processes, which govern microstructural stability and strength of single crystal superalloys. Elementary dislocation processes, which govern low temperature (<800°C) and high stress (>600MPa) creep were studied combining miniature specimen

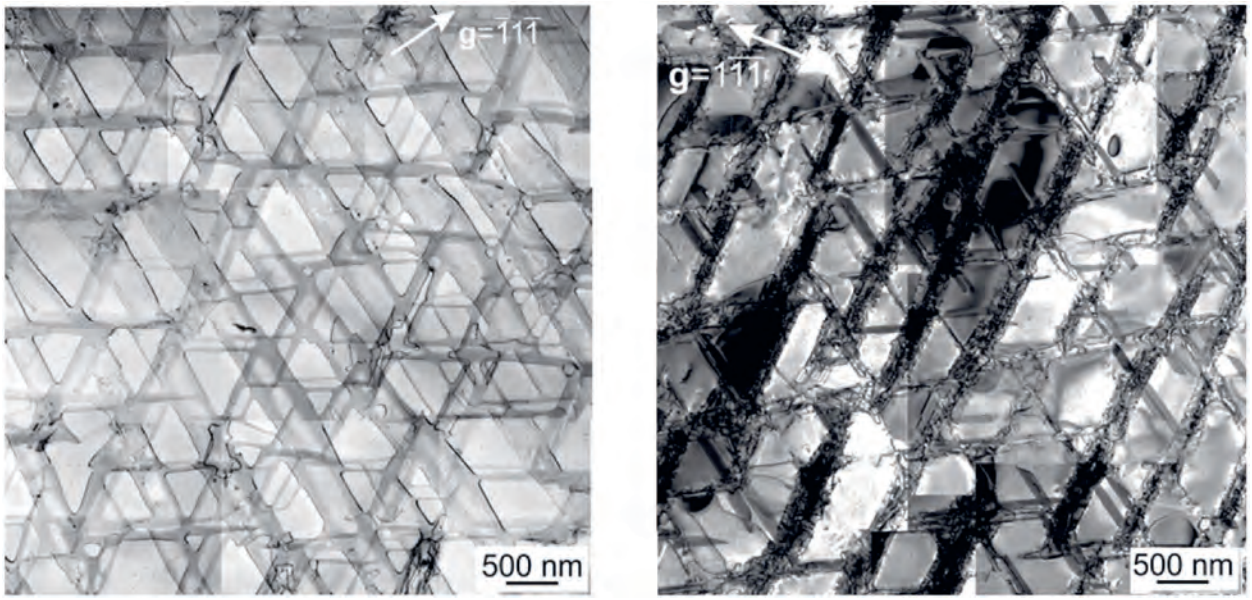


Fig. 1: *STEM montages showing dislocation structures after 0.1 (left image) and 0.4% (creep deformation) at 750°C and 800 MPa. TEM foils parallel to a {111} planes. The STEM montages show how dislocations enter the γ-channels. Dislocations must fill the γ-channels before γ'-cutting by planar faults can occur. For details see [1,2]. Single crystal superalloy: ERBO 1 (CMSX4 type).*

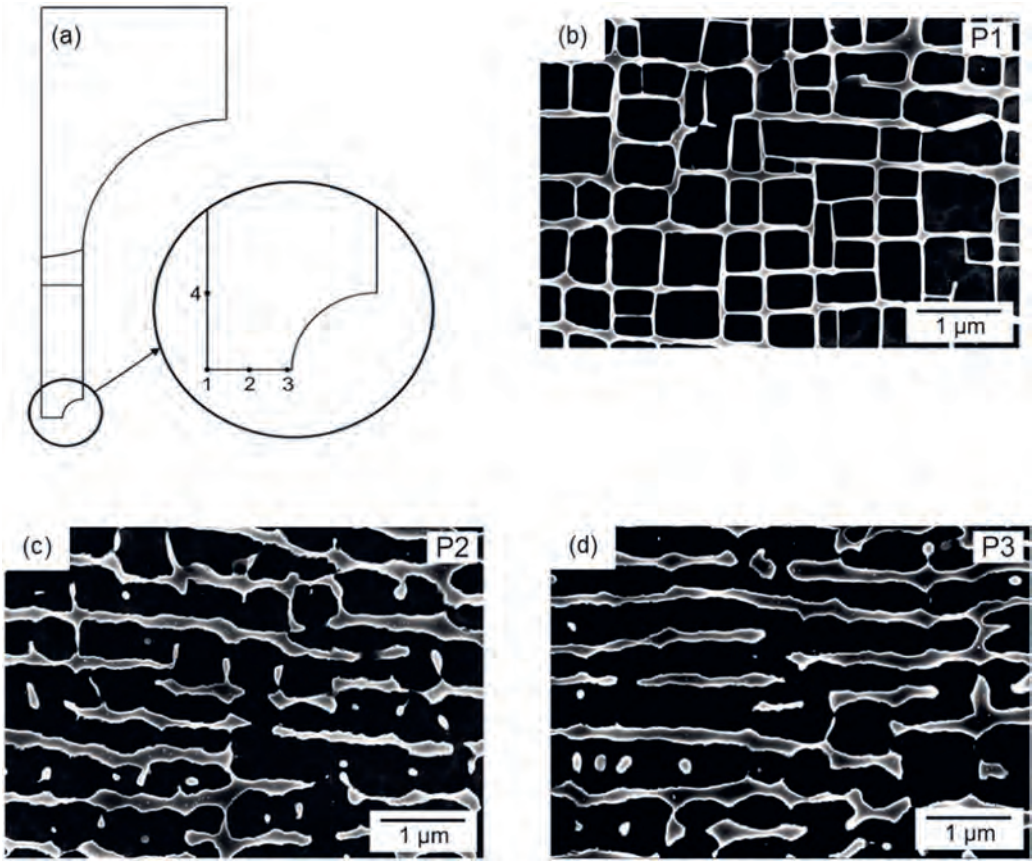


Fig. 2: *Heterogeneous microstructure evolution at different radial positions of a circular notched miniature tensile specimen (net section stress: 300 MPa, temperature: 950°C, time of creep exposure: 169 hours). (a) Schematic illustration of specimen (positions P1, P2 and P3 in the notch root cross-section are highlighted). (b), (c) and (d) SEM micrographs showing the γ/γ'-microstructures at positions P1, P2 and P3 as indicated. For details see [4,5]. Single crystal superalloy: ERBO1 (CMSX4 type).*

creep testing with diffraction contrast transmission electron microscopy [1,2]. The effects of defects on local thermodynamic and kinetic equilibria were investigated [3]. Recently the influence of stress state on the directional coarsening of the γ'-phase (rafting), the well known microstructural evolution of the γ/γ'-microstructure under high temperature creep conditions, was studied [4,5].

References

1. Wu, X.; Wollgamm, P.; Somsen, C.; Dlouhy, A.; Kostka, A.; Eggeler, G.: Acta Mater 112 (2016) 242.
2. Wu, X.; Dlouhy, A.; Eggeler, Y.M.; Spiecker, E.; Kostka, A.; Somsen, C.; Eggeler, G.: Acta Mater 144 (2018) 642.
3. Wu, X.; Makineni, S.K.; Kontis, P.; Dehm, G.; Raabe, D.; Gault, B.; Eggeler, G.: Materialia, available online since September 19th 2018, https://doi/org/10.1016/j.mtla.2018.09.018.
4. Cao, L.; Buerger, D.; Wollgramm, P.; Neuking, K.; Eggeler, G.: Mat Sci Eng A 712 (2018) 223.
5. Cao, L.; Wollgramm, P.; Buerger, D.; Kostka, A.; Cailletaud,G.; Eggeler, G.: Acta Mater 158 (2018) 381.

Max Planck Partner Group on Designing Damage Tolerant Functional Oxide Nanostructures

Group Head: B. N. Jaya

Indian Institute of Technology Bombay, Powai, India

The group aims at micro-mechanical characterization of functional oxide structures in order to design and develop architectures with increased damage tolerance. In cooperation with N.G. Mathews from the Indian Institute of Technology Bombay and C. Kirchlechner and G. Dehm from the SN department, the damage tolerance of Pb-free Barium Titanate (BTO) is studied at small length scales. BTO is chosen as the model material system for its excellent ferroelectric and piezoelectric properties. The effect of specimen size on yield/failure strength, and slip activity in BTO, impact of domain type, as well as its fracture toughness at small length scales has not been reported so far. The following are preliminary results from experiments on BTO Single Crystal (SX).

The BTO SX micropillar compression studies showed a prominent size effect in yield strength (156% increase) while also displaying duplex slip in samples of 2, 1 and 0.5 µm diameter. Micro-cantilever fracture toughness tests reveal K_{IC} = 1.9 MPam$^{1/2}$, 120% higher than those reported earlier using indentation fracture tests at macro-length scales. On the whole, BTO displays enhanced damage tolerance at small length scales. It was also observed that the interference of the electron beam during *in situ* testing does not have any impact on the load-displacement response of BTO SX. This work will be further extended to polycrystalline BTO thin films and their electro-mechanical response will be used to propose strategies to enhance the damage tolerance of such nanostructures.

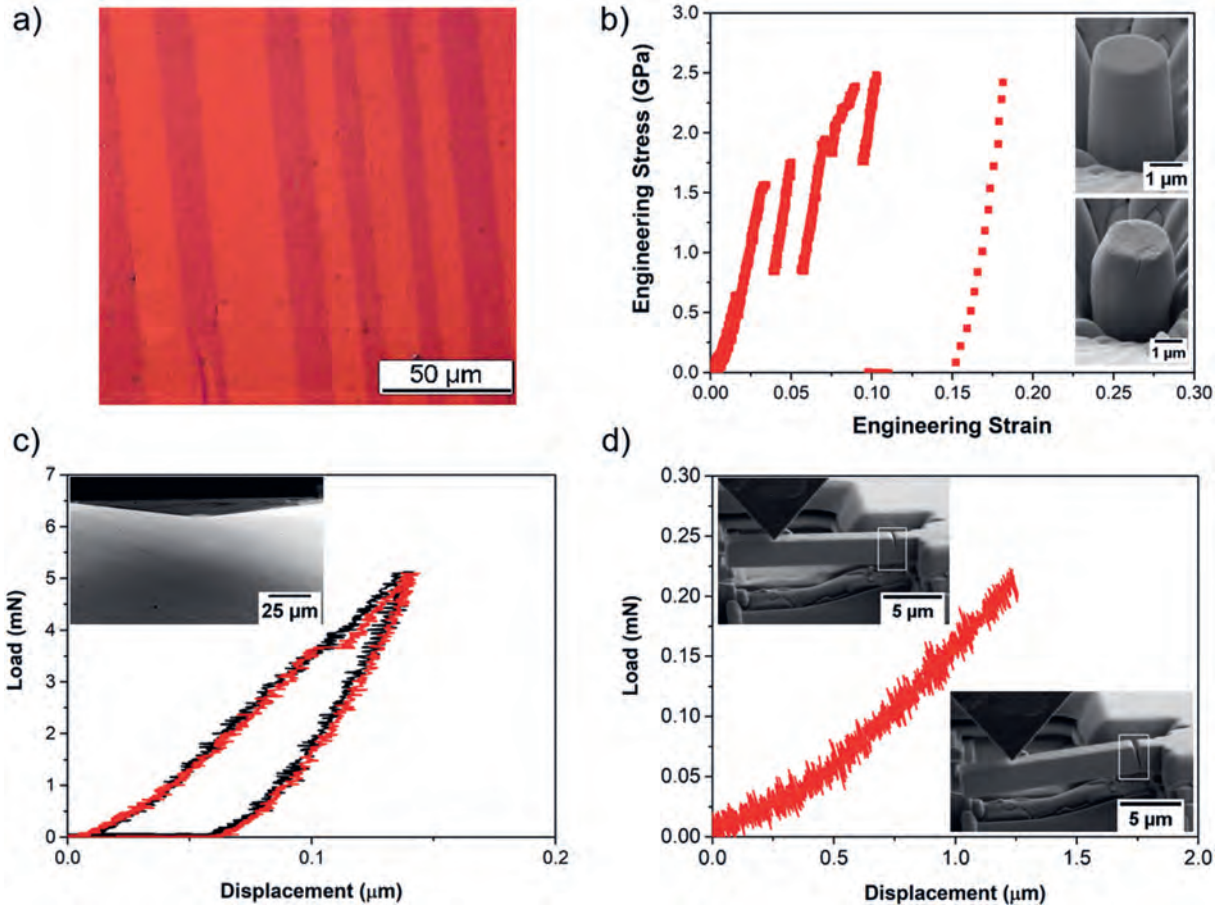


Fig. 1: *a) BTO SX under polarized light showing two different ferroelectric domains, b) Micro-pillar compression experiment on BTO SX showing several load drops corresponding to slip activity, c) Nanoindentation tests on a single domain of BTO SX with the electron beam switched on (black curve) and off (red curve) revealing no difference in behaviour, d) Micro-cantilever fracture test showing linear elastic behaviour with catastrophic fracture.*

Max Planck Partner Group on Combinatorial Design of Novel Rare-Earth Free, High-Entropy Based Permanent Magnets

Group Head: P. Gokuldoss

Indian Institute of Technology, Madras, India

Magnetic materials are used to manufacture essential parts of any electrical motor, generator and transformer, finding application in a wide-range from power plants, house hold appliances to automobiles, industrial and commercial sectors. Most of these electrical machines require high performance permanent magnets that can deliver the necessary power. Currently, only rare-earth based permanent magnets are used for such applications, as the rare-earth elements provide the required features for permanent magnetic response, namely, uniaxial magnetic anisotropy. Hence, intense research has been devoted to developing highly-efficient permanent magnets that are either rare-earth free or require only minor additions in order to reduce the cost without compromising energy efficiency.

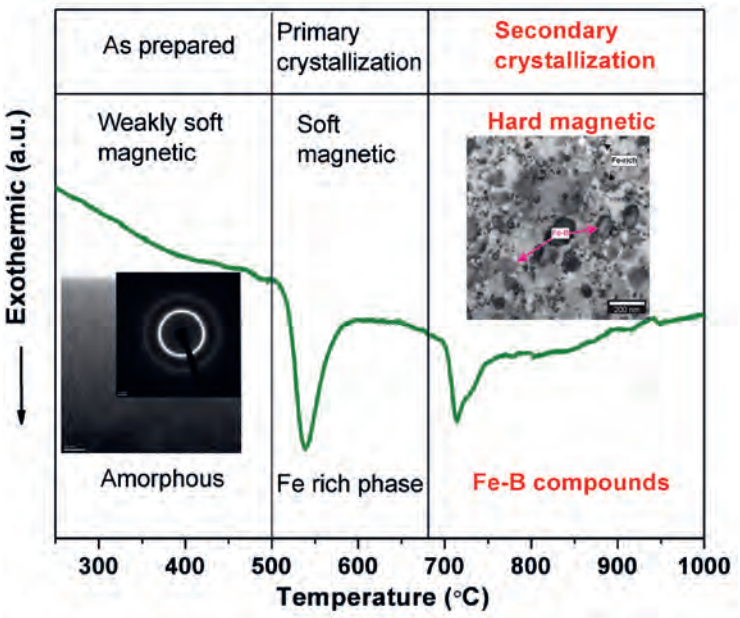


Fig. 1: *Phase evolution in rapidly quenched Fe-B based alloys upon heat treatment.*

Development of new rare-earth free permanent magnets has not been so successful till now considering that the phases that can exhibit uniaxial magnetic anisotropy together with high coercivity are difficult to be identified. Most often these phases are located in the compositional centre of phase diagrams within a limited composition range and are stable only in small temperature intervals. Hence, to identify new phases that can suitably be developed into novel high-performance permanent magnets requires a combinatorial approach to alloy design where extensive composition ranges can be screened experimentally within a short time frame and with limited materials expenditure.

This newly launched project thus combinatorially explores multi-component systems based on the concept of high entropy alloys (HEAs). This concept enables experimentally probing compositions that are multi-component in nature and are also located in the middle of phase diagrams. By employing multiple methods of combinatorial screening such as rapid alloy prototyping, combinatorial thin film deposition, reaction sintering, both equilibrium and non-equilibrium phases can be found and identified for their crystal structures and their magnetic properties as a function of composition.

Max Planck Partner Group on Stress and Defects Driven Phase Transformations

Group Head: S. Meka

Indian Institute of Technology, Roorkee, India

This partner group (PG) was awarded at the end of 2016 and has formally started its activities from 1st March 2017. It is carrying out research activities independently as well as in collaboration with the MPIE. The focus of the research group is to investigate phase transformation phenomena that occur as a result of metal-gas reactions, which, in turn, generate stresses and lattice defects in the material. Some examples of the research are given below.

Researchers from the MPIE and IIT Roorkee are working jointly on developing nitride dispersion strengthened steels by combining the gaseous nitriding expertise of researchers at IIT Roorkee and the Additive Manufacturing (AM) expertise at the MPIE. In this project, Fe-2.3 wt.% Al alloy powder was subjected to gaseous nitriding treatment to realize the internal precipitation of AlN in ferrite matrix in addition to the development of iron nitrides. After that, the developed iron nitrides were reduced to iron and N_2 gas by hydrogen reduction treatment such that AlN particles remain stable, whereas iron nitrides are unstable. The nitrided-plus-hydrogen-reduced powders were consolidated using spark plasma sintering (SPS). Sintered compacts have indicated a significant increase in hardness as compared to the case of no AlN dispersion was created, i.e., SPSed as received Fe-2.3wt.%Al alloy powder. Detailed characterization revealed a complex microstructure beyond the targeted AlN particles in ferrite matrix: During nitriding, iron-oxides have developed which also got reduced during following hydrogen-reduction treatment. Due to the availability of this oxygen, Al-oxynitrides have developed as layers in powder particles. We will further optimize the process to avoid oxygen contamination in the future. It is also planned to carry out ballmilling of Fe-2.3wt.%Al alloy powder to reduce the particle size and to introduce dislocations which will act as nucleation sites for AlN and thus are expected to refine the particles. The final aim of this project is to use the nitrided and hydrogen-reduced powders in additive manufacturing employing selective laser melting (SLM) to realize AlN dispersion strengthened steel components.

Other research activity which the PG is currently focussing on is to understand gas/solid equilibria, especially to understand the possible establishment of metastable states leading to spinodal decomposition of the system. In this context, iron-based alloys which show the possibility of spinodal decomposition during the process of equilibrating with externally imposed gaseous nitriding and oxidising atmospheres are being explored. Further joint work is underway on the effect of nitride inhibitor particles on secondary recrystallization in electrical steels.

Under this PG activity, researchers from the MPIE had visited IIT Roorkee for scientific discussions on the ongoing collaborative research activities and also to conduct a one day workshop on “microstructural aspects of additive manufacturing” at IIT Roorkee. Additional research stays of members of the PG at the MPIE are planned for 2019.

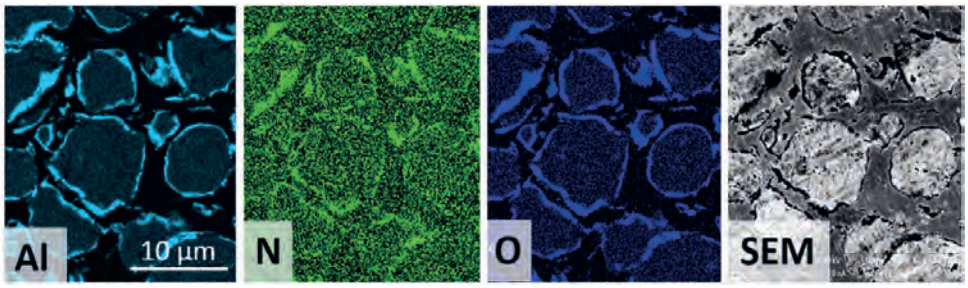


Fig. 1: *Microstructure of SPS-sintered Fe-2.3Al powder that had been gas nitrided and hydrogen reduced prior to sintering. A shell of Al-rich oxynitrides around the previous powder particles is visible.*

References

1. Akhlaghi, M.; Meka, S.R.; Jägle, E.A.; Kurz, S.J.B.; Bischoff, E.; Mittemeijer, E. J.: Metall Mater Trans A 47 (2016) 4578.
2. Sasidhar, K.N.; Kumar, A.; Meka, S.R.: Aluminium Partitioning Triggered by Inward Diffusing Carbon During Carburization of Fe2.3wt%Al Alloy, 14th International Conference on Diffusion in Solids and Liquids (DSL-2018), June 25-29 (2018) Amsterdam, The Netherlands.
3. Yadav, A.S.; Singh, J.; Meka, S.R.: Carburization Induced Phase Transformation in Fe-2wt.% Mn Alloys, 14th International Conference on Diffusion in Solids and Liquids (DSL-2018), June 25-29 (2018) Amsterdam, The Netherlands.
4. Meka,S.R.; Schubert, A.; Bischoff, E.; Mittemeijer E.J.: Development of Nitrides of Silicon and Iron upon Nitriding Fe-Si alloys, European Conference on Heat Treatment (ECHT 2018), April 12-13 (2018) Friedrichshafen, Lake Constance, Germany.

New Research Groups

Materials Science of Mechanical Contacts

Group Head: M. Herbig

Department of Microstructure Physics and Alloy Design (MA)

The non-permanent research group ”Materials Science of Mechanical Contacts” was established in January 2017 with the financial support of the Bundesministerium für Bildung und Forschung (BMBF Junior Research Group on “Material Science for Alternative Energy”). It aims at deepening our understanding of the materials science phenomena associated with intense joint mechanical and environmental contacts. This is key to revealing a broad range of phenomena required for improving crucial engineering components such as bearings, rails, hip implants, extrusion tools, boring heads, cutting inserts or dental fillings.

One main focus of this research group is the study of white-etching-cracks (WECs), which are primarily known to cause failure in bearings and rails, but which are in reality ubiquitous in high carbon steel applications subjected to intense mechanical contacts. This failure mode causes billions of euros costs worldwide each year. The group follows a diversified approach to yield a breakthrough on this long-standing challenge: Specimen failure under controlled laboratory conditions is generated using a customized rolling contact fatigue machine built in-house that simulates test conditions similar to ball bearings but on self-designed alloys. Both, lubrication and loading conditions as well as the electric current flow through the bearing (which is of importance as electric discharge events have been associated with the presence of WECs) can be controlled with this instrument. These specimens are compared to those failed in service using state-of-the-art microscopy. Individual phenomena are investigated separately where possible. Dedicated experiments are conducted to investigate the mechanisms of precipitate decomposition by deformation, heat and electricity. A further project aims at the direct observation of solute hydrogen by atom probe tomography (APT) in deformed pearlite to shed light on the role of hydrogen in the process of cementite decomposition. The fracture toughness of white-etching-layers in rails is determined by cantilever bending tests in the scanning electron microscope.

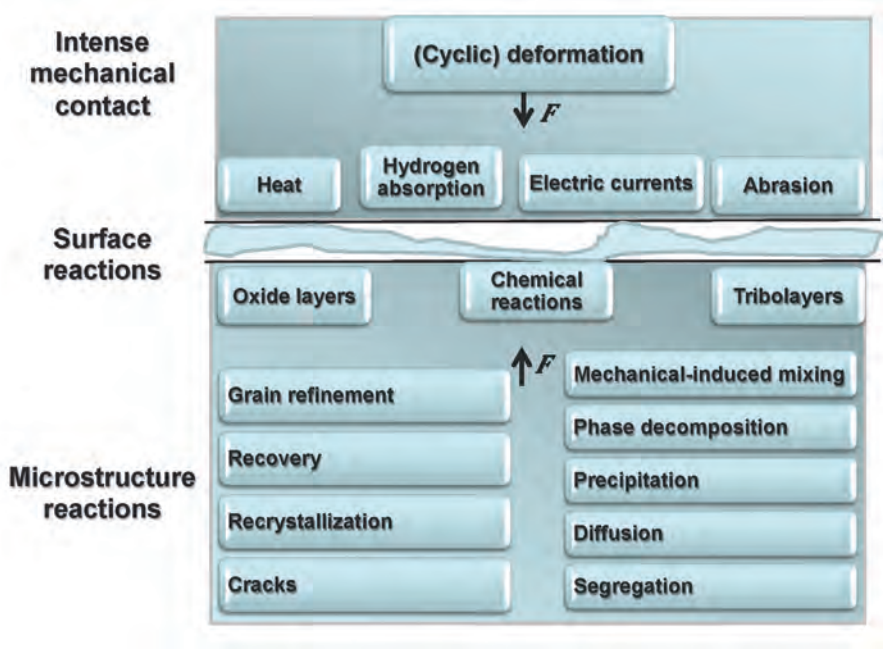


Fig. 1: *Microstructure reactions caused by intense mechanical/environmental contacts.*

The contact between two solid bodies subjected to high forces under harsh environmental conditions and large numbers of repetitions involves complex materials science phenomena (Fig. 1): Plastic deformation can lead to fatigue, grain refinement and precipitate decomposition. Frictional heat can cause diffusion, phase transformation, recovery or recrystallization. The presence of air, lubricants or body fluids at the contact point causes oxidation, tribolayers, or even corrosion or hydrogen embrittlement. These processes usually occur simultaneously in service and cannot be tracked *in situ*. The analysis of such phenomena requires combined chemical and structural characterization down to the atomic scale.

The research activities on steels are complemented by research on hip implants where corrosion and wear debris at the contact point between the stem and head leads to adverse tissue reactions, requiring the explantation of 2.5% of all prostheses. Here, correlative transmission electron microscopy and APT give access to the complex body/implant interactions.

Complex Concentrated Alloys

Group Head: F. Körmann

Department of Computational Materials Design (CM)

The Complex Concentrated Alloys (CCA) research group of Fritz Körmann, established in 2018, aims at the computational design and accelerated exploration of multi-component alloys with parameter-free (*ab initio*) computer simulations [1]. A main focus is currently on complex concentrated alloys and related concepts such as multi-principal element alloys and high-entropy alloys. Mechanical [2,3], magnetic [4], thermodynamic [5,6] and other materials properties [7-9] are investigated by means of computer simulations in close collaboration with experimental and theoretical partners at the MPIE and worldwide.

Multicomponent alloys with multiple principal elements including high-entropy alloys and compositionally complex alloys are attracting rapidly growing attention. The endless possibilities to explore new alloys and the hope for better combinations of materials properties have stimulated a remarkable number of research works in the last years. Most of these works have been based on experimental approaches, but *ab initio* calculations have emerged as a powerful approach that complements experiment and serves as a predictive tool for the identification and characterization of promising alloys [1].

The CCA group focuses on the theoretical *ab initio* modelling of phase stabilities and materials properties of multi-principal element alloys by means of density functional theory (DFT) [1]. Concepts and methods are developed to efficiently consider chemical disorder [1-9] and various finite-temperature excitations [3-6,8,9] based on DFT. Different concepts to study crystal and alloy phase stabilities [2,3,5,6], the impact of lattice distortions [7], magnetic transitions [4], and chemical short-range order [6] are objectives of the group. Strategies to study elastic properties, stacking fault energies, and their dependence on, e.g., temperature or alloy composition are evaluated in close collaboration with other departments at the MPIE.

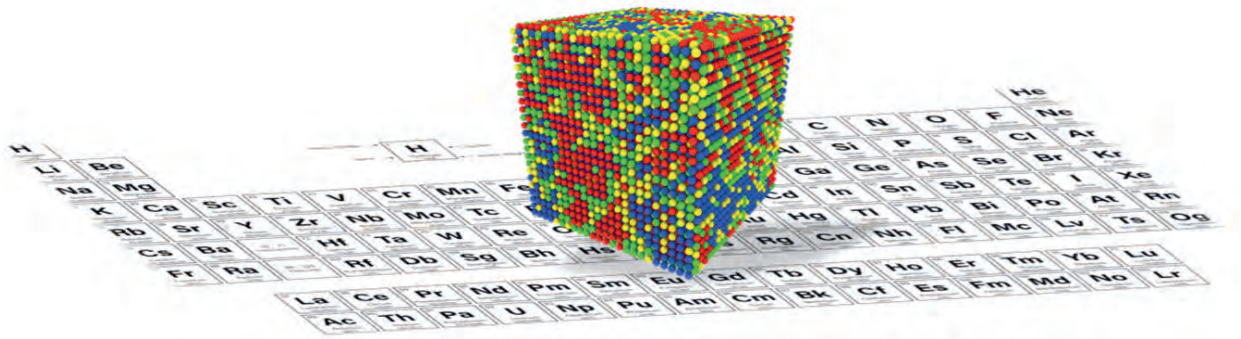


Fig. 1: *Sketch of a four-component high entropy alloy. A key aim of the CCA group is to develop the computational methods for screening the large compositional phase space of such multi-component alloys for phase stabilities and outstanding materials property combinations.*

References

1. Ikeda, Y.; Grabowski, B.; Körmann, F.: Mater Charact (2018).
2. Rogal, L.; Bobrowski, P.; Körmann, F.; Divinski, S.; Stein, F.; Grabowski, B.: Sci Rep-UK 7 (1) (2017) 2209.
3. Li, Z.; Körmann, F.; Grabowski, B.; Neugebauer, J.; Raabe, D.: Acta Mater 136 (2017) 262.
4. Körmann, F.; Ma, D.; Belyea, D.D.; Lucas, M.S.; Miller, C.W.; Grabowski, B.; Sluiter, M.H.F.: App Phys Lett 107 (2015) 142404.
5. Körmann, F.; Sluiter, M.H.F.: Entropy 18 (2016) 403.
6. Körmann, F.; Ruban, A.V.; Sluiter, M.H.F.: Mater Res Lett 5 (1) (2017) 35.
7. Oh, H.S.; Ma, D.; Leyson, G.P.; Grabowski, B.; Park, E.S.; Körmann, F.; Raabe, D.: Entropy 18 (9) 321 (2016) 321.
8. Körmann, F.; Ikeda, Y.; Grabowski, B.; Sluiter, M.H.F.: npj Comput Mat 3 (2017) 36.
9. Ikeda, Y.; Körmann, F.; Dutta, B.; Carreras, A.; Seko, A.; Neugebauer, J.; Tanaka, I.: npj Comput Mat 4 (2018) 7.

High-Entropy Alloys

Group Head: Z. Li

Department of Microstructure Physics and Alloy Design (MA)

Funded by the German Research Foundation (DFG), this new group was established in 2017 with the aim to develop novel high-entropy alloys (HEAs) with exceptional mechanical, physical and chemical properties based on the understanding of their structure-properties relations.

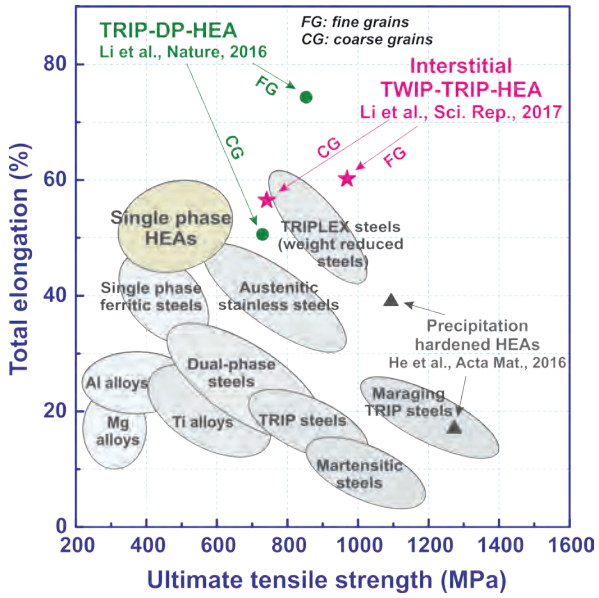


Fig. 1: *Strength-ductility profiles of various classes of metallic materials including the new HEAs developed by the group.*

Conventional alloy design over the past centuries has been constrained by the concept of one or two prevalent base elements. As a breakthrough of this restriction, the concept of HEAs opens a new realm of numerous opportunities for investigations in the huge unexplored compositional space of multi-component alloys.

While conventional alloys use strengthening mechanisms such as grain boundaries, dual-phase (DP) structure, dislocation interactions, precipitates and solid solution (e.g. steels, Ti-alloys, Al-alloys), the novel interstitial TWIP-TRIP-HEAs (TWIP: Twinning-induced plasticity; TRIP: Transformation-induced plasticity) concept developed by this new research group combines all available strengthening effects, namely, interstitial and substitutional solid solution, TWIP, TRIP, multiple phases, precipitates, dislocations, stacking faults and grain boundaries. This leads to the exceptional strength-ductility combination of the novel HEAs, exceeding that of most metallic materials.

The research group conducts the state-of-the-art research work employing novel experimental methodologies (Fig. 2), e.g., electron backscatter diffraction (EBSD), electron channeling contrast imaging (ECCI), atom probe tomography (APT) and transmission electron microscopy (TEM), in the following specific aspects:

- Excellent strength-ductility combination of transitional metal HEAs
- Resistances to hydrogen-embrittlement and corrosion of HEAs
- Light-weight high-strength HEAs
- High-temperature refractory high-strength HEAs
- Multifunction of HEAs
- Defects, segregations and thermodynamics in HEAs
- *In situ* observation of deformations in HEAs under electron microscopes

These aspects are strongly interconnected and facilitate an extensive collaboration network with national and international experts.

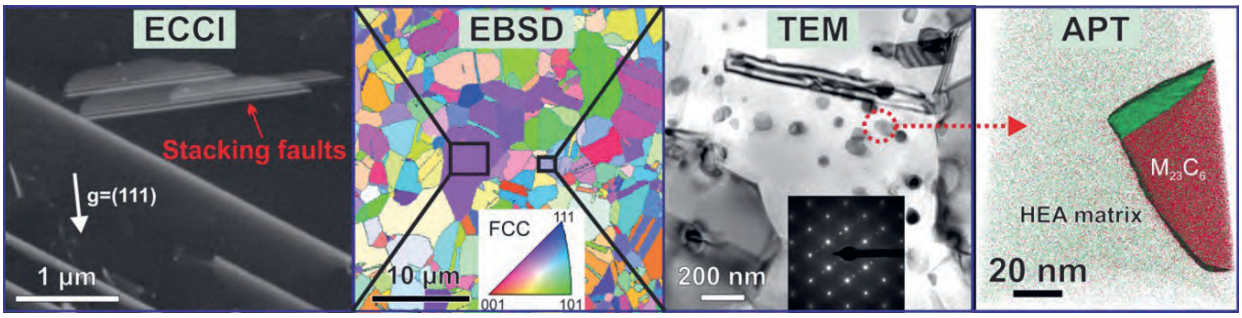


Fig. 2: *Representative microstructural information obtained from an interstitial TWIP-TRIP-HEA sample by combining multiple advanced characterization techniques.*

References

1. Li, Z.; Pradeep, K.G.; Deng, Y.; Raabe, D.; Tasan, C.C.: Nature 534 (2016) 227.
2. Li, Z.; Körmann, F.; Grabowski, B.; Neugebauer, J.; Raabe, D.: Acta Mater 136 (2017) 262.
3. Lu, W.; Liebscher, C.H.; Dehm, G.; Raabe, D.; Li, Z.: Adv Mater (2018) 1804727.
4. Su, J.; Raabe, D.; Li, Z.: Acta Mater 163 (2019) 40.

New Scientific Laboratories and Facilities

The LAPLACE Project: Preparation and Transfer of Specimens for Electron Microscopy and Atom Probe Tomography under Ultra-High Vacuum and Cryogenic Conditions

L.T. Stephenson [1], U. Tezins [1], A. Sturm [1], D. Vogel [2], Y. Chang [1], W. Lu [3], M. Rohwerder [2], D. Raabe [1], B. Gault [1]

[1] Department of Microstructure Physics and Alloy Design (MA)
[2] Department of Interface Chemistry and Surface Engineering (GO)

The Atom Probe Tomography (APT) group in the MA department is developing integrated protocols for ultra-high vacuum cryogenic specimen transfer between platforms without exposure to atmospheric contamination. This apparatus enables back-and-forth transfers that will allow precise imaging of the specimen at various stages of the analysis. In theory, recording the emitter shape could provide input to improve APT data reconstruction protocols, and a future research direction will be to push for chemical compositional mapping with true atomic resolution. This explains the allusion to the 'Laplace demon', which refers to the statement by Laplace that by knowing the position and nature of all particles in the universe at a given point in time, one could predict the future and know all of the past.

Operating as of May 2018 are four linked platforms; a N_2-atmosphere glovebox, two state-of-the-art atom probes, and a scanning electron microscope / Xe-plasma focused ion beam equipped with a cryo-stage (funded by the federal ministry BMBF). It will be complemented with a small reaction chamber to allow for gas charging and surface reactions induced directly on atom probe specimens, which is developed together with the GO department and planned to be installed mid 2019.

Up until now, the outcome of the UHV specimen transfer has allowed the analysis of environmentally-sensitive materials by atom probe tomography, as well as, through cryogenic preservation, specimens that have been specifically modified by chemical reactions at their surface, i.e. deuterium or hydrogen charging. We demonstrated the efficacy of the new protocols by the successful preparation and transfer of delicate samples. First, we prepared a series of specimens from commercially pure Mg and Ti, as well as specimens from a Ti-6Al-2Sn-4Zr-6Mo alloy. Mg is highly sensitive to oxidation and we demonstrated that a thin layer of surface oxide was formed in the high-vacuum chamber of the plasma focussed ion beam instrument [1]. Commercially pure Ti and Ti-alloys are prone to forming hydrides during preparation as shown in Fig. 2. Here, we showed how maintaining the specimen at low temperature during the final stages of sharpening of the needle-shaped specimen could alleviate this issue. The results from APT displayed in Fig. 2 were also confirmed at a larger scale via transmission Kikuchi diffraction and transmission electron microscopy [2]. This result is critical for the success of the ERC-Consolidator project of B. Gault (see p. 70) that aims to precisely measure the H-content in engineering materials to explain their failure in service.

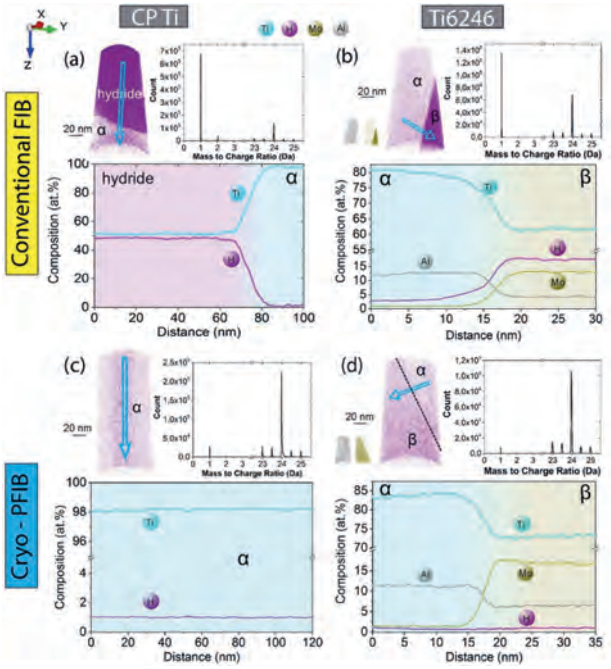


Fig. 1: *H distribution map obtained from APT, mass spectrum and composition profile of (a) commercially-pure titanium and (b) a Ti-6Al-2Sn-4Zr-6Mo alloy from specimens prepared by conventional focused ion beam at room temperature; (c)-(d) specimens from similar materials prepared by cryo-plasma focused-ion beam (at approx. -140°C).*

The Laplace project provides unique, state-of-the-art infrastructure for investigations in materials science and also allows for exploring new opportunities. We also demonstrated that the preparation and transfer of ice, which we hope will be an effective carrying medium for analysing suspensions and e.g. nanoparticles, and we recently successfully analysed proteins fibrils [3] involved in Alzheimer disease.

References

1. Stephenson, L.T.; Szczepaniak, A.; Mouton, I.; Rusitzka, K.; Breen, A.J.; Tezins, U.; Sturm, A.; Vogel, D.; Chang, Y.; Kontis, P.; Rosenthal, A.; Shepard, J.D.; Maier, U.; Kelly, T.F.; Raabe, D.; Gault, B.: PLoS ONE 13(12): e0209211.
2. Chang, Y.; Lu, W.; Guénolé, J.; Stephenson, L.; Szczpaniak, A.; Kontis, P.; Ackerman, A.; Dear, F.; Mouton, I.; Zhong, X.; Zhang, S.; Dye, D.; Liebscher, C.H.; Ponge, D.; Korte-Kerzel, S.; Raabe, D.; Gault, B.: Nat Comm (2019) accepted.
3. Rustiska, K.; Stephenson, L.T.; Szczepaniak, A.; Gremer, L.; Raabe, D.; Willbold, D.; Gault, B.: Sci Rep (8) (2018) 17615.

Additive Manufacturing and Powder Synthesis Laboratory

E. A. Jägle

Department of Microstructure Physics and Alloy Design (MA)

In the group "Alloys for Additive Manufacturing", we perform research to develop alloys that are suitable for use in additive manufacturing (AM). This can mean that they are resistant to typical processing-induced defects such as hot tearing. It can also mean that they exploit particular characteristics of the AM process. The three features of AM that our group focusses on are:

1. the high cooling rates of up to 10^6 Ks^{-1} after deposition,
2. the powder-technological nature of the process and
3. the possible metal-gas interactions occurring during material build-up.

Fig. 1: *Image of the SLM process during operation (left). The "sparks" captured are the typical ejected powder particles that are carried away from the melt pool by the inert gas cross jet. On the right hand side, the gas atomization process is shown (initial experiments).*

To investigate the impact of these features and to be able to develop new alloys, we aim to cover the entire process chain of additive manufacturing in-house. This starts with the design of alloys using thermodynamic databases, in particular using the software Thermocalc. Next, the alloys of interest are cast in rod shape by the metallurgy group. We have designed and built a laboratory-scale gas atomizer that enables us to produce small batches of powder from the cast rods. The powder is then used in our Aconity 3D "Mini" Selective Laser Melting (SLM) research machine to produce specimens which are analysed with the Institute's extensive testing and characterization facilities. We also pursue two other alloy development routes. In the first screening stage, small coupons are produced by arc melting and re-melted in the SLM machine. In the second, the alloy is generated during the process by melting a mixture of powders.

The devices available in the laboratory are designed or selected, to optimally support the research group's mission: To efficiently design and produce new alloys specifically for AM. There are very few atomizers available worldwide in research institutes that are capable to produce small powder batches of just a few hundred grams in the required size range. The atomizer is of the EIGA-type (Electrode Induction Melting Gas Atomization), which enables us to process reactive and refractory metals. Additionally, it is built in a modular fashion, allowing for easy cleaning between experiments and future modifications.

Among the available AM processes, we focus our work on the SLM process because it involves the smallest melt pools and hence the highest cooling rates. Our SLM machine is equipped with a small build platform and therefore requires only little powder. Its build platform heating system allows processing materials up to 800°C, which gives a high degree of freedom to avoid defects and to perform *in situ* heat treatments. In the beam path of the laser, there are two high-speed spot pyrometers for process monitoring. Finally, also this machine is designed for simple powder exchange and minimum cross-contamination.

In addition to the larger machines, the laboratory includes some powder handling equipment such as a sieving station, a powder drying oven, high-energy ball mills as well as a microbalance with a density measurement kit.

References

1. Bajaj, P.; Wright, J.; Todd, I.; Jägle, E.A.: Additive Manufacturing (2018) in press. DOI: 10.1016/j.addma.2018.12.003.
2. Makineni, S.K.; Kini, A.R.; Springer, H.; Raabe, D.; Gault, B.: Acta Mater 151 (2018) 31.
3. Chauvet, E.; Kontis, P.; Jägle, E.A.; Raabe, D.; Tassin, C.; Blandin, J.J.; Dendievel, R.; Vayre, B.; Abed, S.; Martin, G.: Acta Mater 142 (2018) 82.
4. Chang, Y.; Breen, A.J.; Trazimoghadam, Z.; Kürsteiner, P.; Gardner, H.; Ackerman, A.; Radecka, Bagot, P.A.J.; Lu, W.; Li, T.; Jägle, E.A.; Herbig, M.; Stephenson, L.T.; Moody, M.P.; Rugg, D.; Dye, D.; Ponge, D.; Raabe, D.; Gault, B.: Acta Mater 150 (2018) 273.
5. Jägle, E.A.; Sheng, Z.; Lu, L.; Wu, L.; Risse, J.; Weisheit, A.; Raabe, D.: JOM 68 (2016) 943.
6. Jägle, E.A.; Sheng, Z.; Choi, P.; Raabe, D.: Proceedings of the International Conference on Solid-Solid Phase Transformations in Inorganic Materials (2015) 1029.

3D-Orientation Microscopy by Automated Mechanical Polishing Combined with EBSD and ECCI

S. Zaefferer [1], P. Konijnenberg [1,2], S.-P. Tsai [1], I. Gonzalez [3], S. Hartke[1]

[1] Department of Microstructure Physics and Alloy Design (MA)
[2] Bruker Nano
[3] Electronics Group

3D orientation microscopy (3D-ORM) based on electron backscatter diffraction (EBSD)-mapping and serial sectioning is a powerful and versatile tool to obtain insights into the 3-dimensional nature of microstructures. 3D-ORM offers basic morphological data on microstructures, like the 3-dimensional shape and size of crystals, the arrangement of crystals and phases (e.g. neighbourhood or interconnectivity of structures), the 3-dimensional distribution of precipitations, dislocations and the extension and magnitude of elastic and plastic strain fields. The technique should be employed in cases where microstructures cannot be inferred through stereological approaches from sample sections. A particular information, which, in most cases, cannot be inferred in this way is the crystallographic and morphological character of grain and phase boundaries, which, however, determines the physical and chemical properties of these highly important lattice defects. The crystallographic character is described by 5 parameters, namely the misorientation across the boundary (3 parameters) and the boundary plane normal vector (2 parameters). The morphology may be described, for example, by local curvature or faceting of the interface.

Both, the morphological and the crystallographic character, may vary strongly even along one single grain or phase boundary, which means that they need to be determined locally if properties like mobility, corrosion or light emission (in the case of semiconducting materials) are to be explained. So far, 3D-ORM based on serial sectioning by focused ion beam milling (FIB) has been developed at the MPIE and other places worldwide. This technique has the advantage of being easily automatable and delivering precisely spaced sections. Its disadvantages, however, are the severely limited volume that can be investigated with the technique (usually about 20 x 20 x 20 µm³), the inconvenient geometry of the investigated volume, and the often significant beam damage caused by the high-energy ion beam milling. As a consequence we have looked into other techniques of serial sectioning which may be broad ion beam milling, laser ablation and mechanical-chemical surface polishing. For a number of reasons we decided to invest into the mechanical-chemical polishing approach which offers very large volumes to be investigated (in the order of 1000 x 1000 x 100 µm³ or even larger), in principle leads to ideal surface quality with a minimum of damage, and results in ideally flat surfaces which are very well suited for further measurements of, e.g., corrosion or optoelectronic properties. The challenge with this approach is, however, the difficulty of automation and the exact measurement and control of section thicknesses. To overcome these problems we have bought a polishing robot (Saphir XChange, Fa. ATM) and a “Cobot” (i.e. a collaborative robot) (UR5, Universal Robots). Both instruments are connected to a Zeiss Merlin scanning electron microscope with an EBSD and energy dispersive x-ray spectroscopy (EDX) system. The Saphir XChange polishes a sample in a perfectly controlled manner, the UR5 transports the sample to and from the microscope and the microscope performs EBSD and electron channelling contrast imaging (ECCI) measurements on the polished sample surface. To integrate these instruments into one 3D-ORM system we have put together a team of 5 people with different backgrounds, including material science, mechanical engineering, electronics and software design. The system is expected to make first complete runs in early 2019.

Fig. 1: *Combination of the Saphir X-Change polishing automata (the main gray box), the UR5 cobot (in the front) and the Zeiss Merlin SEM (behind both in the background). The cobot is currently picking up a sample which has been placed on an exchange table by the polishing automata. It will subsequently open the microscope door, place the sample in the microscope and close the door again.*

Aberration Corrected Transmission Electron Microscopy Facility

C. Liebscher [1], V. Kree [1], J. Thomas [1], G. Dehm [1], C. Scheu [2]

[1] Department of Structure and Nano-/ Micromechanics of Materials (SN)
[2] Max Planck Research Group on Nanoanalytics and Interfaces (NG)

The aberration corrected transmission electron microscopy (TEM) facility was completed with the installation of the image corrected FEI Titan Themis 80-300 (Thermo Fisher Scientific) mid of 2017 illustrated in Fig. 1. Since its commissioning in October 2017, the microscope is available for C_s-corrected TEM imaging with a point resolution of down to 80 pm, enabled by a CEOS GmbH aberration corrector. The microscope can be operated at 80, 200 and 300 kV acceleration voltage. Conventional high-resolution imaging can be coupled with through focal series acquisition for exit wave reconstruction. Low dose conditions can flexibly be adjusted to study beam-sensitive materials and to reduce beam-induced artifacts. The microscope is also equipped with a Lorentz lens for imaging magnetic domain walls in materials. It is also possible to operate the microscope in basic scanning TEM (STEM) mode, which can be coupled with energy dispersive X-ray spectroscopy (EDS) through four synchronized EDS detectors (ChemiSTEM).

Currently, the microscope is upgraded with a high speed CMOS (Complementary Metal Oxide Semiconductor) based camera to enable image acquisition with up to 300 frames per second (fps). This is needed to study dynamic events in materials under *in situ* conditions. Since the beginning of 2017, a Gatan model 654 straining holder is available for the FEI microscopes, which allows to strain micron-sized sample *in situ* in the TEM to observe their deformation behaviour. An image of the holder tip, the copper support and the location of a focus ion beam (FIB) prepared sample are shown in Fig. 1.

In May 2018, a DENSsolutions Lightning heating and biasing holder was implemented that allows to heat micron-sized samples up to temperatures of 1300 ºC, and in addition it is possible to apply high electric fields of up to 100 kV/cm at temperatures up to 800 ºC. The tip of the sample holder with electrical contacts and the MEMS (Micro-Electro-Mechanical System) based chip are illustrated in Fig. 1. The novel MEMS based heating/biasing chip design enables unprecedented sample stability with a drift rate of <0.3 nm/min and strongly reduced bulging of the Si_3N_4 membrane of <500 nm up to 500ºC.

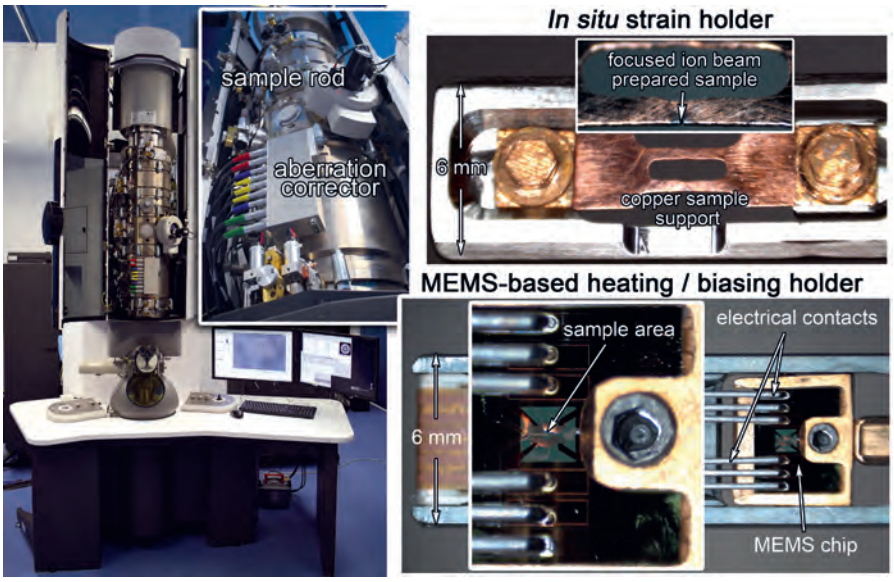


Fig. 1: *Image corrected FEI Titan Themis 80-300 (Thermo Fisher Scientific). The insets highlight the aberration corrector and the sample rod. Close up images of the tips of a Gatan model 654 in situ straining holder and a MEMS based DENSsolutions Lightning heating/biasing holder.*

Hence, the aberration corrected transmission electron microscopy facility enables to investigate the sample behaviour *in situ* under an applied strain, temperature or bias and the dynamic evolution of the micro- and nanostructure can be captured with high-speed electron detectors.

Thermal Analysis Laboratory

F. Stein, L. Christiansen, A. Bobrowski

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

Thermal analysis serves to study physical properties of materials as a function of temperature. The most versatile methods for this purpose are differential thermal analysis (DTA) and differential scanning calorimetry (DSC). In general, DTA/DSC allows the detection of any type of thermal effect in a material occurring during heating or cooling with a constant rate or when holding at constant temperature.

DTA/DSC is applied at the MPIE in all sorts of projects for various types of materials most of metallic but also of ceramic or even biological nature. The available facilities cover a wide range of temperatures from -196 °C (liquid nitrogen) to 2000 °C. Examples for investigated effects comprise melting behaviour, invariant reactions, order-disorder transitions, magnetic transformations, and martensitic transformations in, e.g., steels, but also in shape memory alloys. Moreover, crystallization/recrystallization, relaxation or decomposition processes and glass transitions in amorphous materials can be studied, metastable states can be identified, and precipitation processes can be detected. In addition, the kinetics of effects as for example slow solid-solid transformations or precipitation reactions can be analysed.

Another important aspect of the DSC method is its capability to generate calorimetric data. By exploiting the heat flow signals, heat capacity can be determined between room temperature and about 1200 °C. Finally, the DTA/DSC instruments can also be perfectly well applied for precisely controlled heat treatments to attain special microstructural states while simultaneously recording the heat flow signal.

The DTA/DSC facilities available in our thermal analysis laboratory comprise a low-temperature device 'DSC1' (Co. Mettler-Toledo) capable of measuring in the range from -196 °C (liquid nitrogen) to 600 °C, and two classical DTA/DSC instruments 'DSC 404 C Pegasus' (Co. Netzsch) and 'Setsys 16/18' (Co. Setaram) working between room temperature and 1650 °C and 1750 °C, respectively. Recently, the thermal analysis laboratory was complemented by an instrument for simultaneous thermal analysis 'STA 449 F3 Jupiter' (Co. Netzsch) combining DTA/DSC with thermogravimetric analysis (TGA). For the development of alloys for applications at very high temperatures, e.g. for the replacement of Ni-based superalloys, there is a need to measure the thermal behaviour up to very high temperatures. The STA instrument possesses a graphite furnace allowing DTA experiments up to 2000 °C. Even though the experiments are performed in a He inert gas atmosphere, metallic materials often show a strong sensitivity for chemical reactions with remaining gaseous impurities and often also with the crucible materials. Therefore, simultaneously to recording the DTA signals, weight changes of the sample are monitored by TGA. The STA instrument is equipped with two furnaces, switching from the graphite furnace to the rhodium furnace allows to apply the STA for heat capacity measurements.

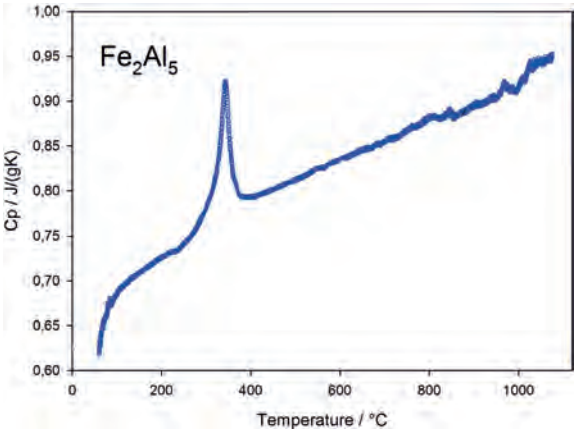


Fig. 1: *The picture on the left side shows the opened STA instrument with DSC sample holder and the two furnaces in the position to switch from the 2000 °C graphite furnace (left side) to the rhodium furnace (right side) suited for heat capacity (C_p) measurements up to 1200 °C and classical DTA/DSC up to 1650 °C; the diagram shows a C_p measurement of a single-phase Fe_2Al_5 intermetallic alloy indicating a structural transformation.*

Long-term Oriented Method Development

Understanding the Complexity of Dislocation Structures using 3-Dimensional Electron Channelling Contrast Imaging (3D-ECCI)

S. Zaefferer [1], P. Konijnenberg [1,2], S.-P. Tsai [1]

[1] Department of Microstructure Physics and Alloy Design (MA)
[2] Bruker Nano

The microstructure of a material, i.e. the ensemble of its extended crystal defects (grain and phase boundaries, precipitations, dislocations and stacking faults, elastic and plastic strain fields) , is intrinsically 3-dimensional (3D), even if many techniques for microstructure characterization (light microscopy, transmission electron microscopy (TEM), scanning electron microscopy (SEM) techniques) reveal only 2D sections or shallow 3D projections of it. Nevertheless, the properties of materials and processes of microstructure formation usually depend strongly on the 3D arrangement of defects. This relation is obvious for grain and phase boundaries for which the local crystallographic and morphological character plays an important role for their properties. Moreover, the 3D arrangement is also important for dislocations and their related stress fields. One interesting and fundamental example is the question whether dislocations that are found inside of deformed grains are produced by activation of Frank-Read sources inside of the grain or whether they originate on the grain boundaries. 2D sections of grains almost never explicitly show the location and nature of dislocation sources. Another example is the distribution of dislocations in front of progressing lenticular martensite crystals. In this case dislocations are important for the arrest of the martensite growth due to the loss of lattice coherency. A third example is the distribution of dislocations along triple junctions in deformed grains and the related formation of new crystals during recrystallization.

The observation and quantification of dislocations has long time been a domain of TEM, which, however, requires thin foil samples and therefore, in most cases, impedes a true 3D observation (except for the 100 nm to 200 nm of volume thickness that is observed by the technique). In contrast, the still relatively new electron channelling contrast imaging (ECCI) technique, applied in SEM, allows observation of dislocations and other defects in the first 100 nm thickness below the surface of bulk samples. If this technique is combined with a sufficiently gentle method to remove thin serial sections of a material, it is possible to perform 3D dislocation observation over large volumes and at a large number of locations in a single sample. An appropriate method for serial sectioning is the mechano-chemical polishing with an oxide particle suspension (OPS) of suitable composition. We are currently building up a fully automated system for this approach (see p. 36). We expect that this system will be able to perform sections of minimum thickness of about 50 nm with a surface quality sufficient for ECCI observations which will allow tracing individual dislocations through a large depth of 10 or more sections.

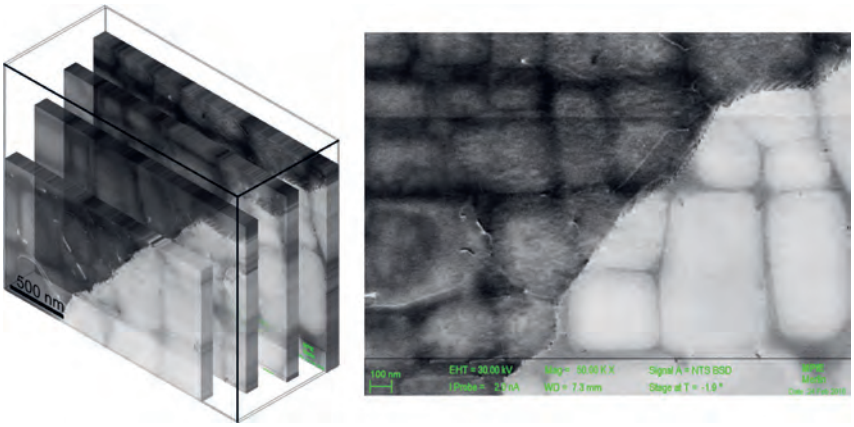


Fig. 1: *3D-ECCI on a subgrain boundary in a single crystal superalloy with a γ-γ' microstructure. The left image shows 4 sections with about 150 nm spacing, displayed as semitransparent ECC images. The right side shows the last section. The cuboidal γ' precipitates are clearly visible. The subgrain boundary consists of a network of narrow-spaced dislocations, visible as white lines. The bright and dark sides are due to different channelling conditions on the two sides of the boundary.*

A first test of this method has been pursued by manual polishing of a superalloy with a γ-γ' microstructure. The figure above shows the arrangement of dislocations along a subgrain boundary in this material. The left side displays the 3D arrangement; the right side shows just the last section. Slice thickness is not yet well controlled and the software for imaging the dislocations is not yet well developed. Both will be subject of future developments.

In situ Electron Microscopy Techniques

C. Liebscher[1], W. Lu[1], S. Lee[1], C. Kirchlechner[1], S. Hieke[2], S. Zhang[2], C. Scheu[2], G. Dehm[1]

[1] Department of Structure and Nano-/ Micromechanics of Materials (SN)
[2] Independent Max Planck Research Group on Nanoanalytics and Interfaces (NG)

In situ electron microscopy offers novel insights into the dynamic evolution of microstructure at elevated temperatures [1-3], high oxygen partial pressures [4], an applied strain [5] or electrical bias at highest possible resolution, even down to the atomic scale. Furthermore, it is possible to probe local chemical and electrochemical reactions of nanomaterials in gaseous and liquid environments [6] by still preserving nanometre resolution. This broad range of *in situ* techniques is currently under development and implementation at the MPIE for both scanning electron (SEM) and transmission electron microscopy (TEM). The aim is to establish methodologies suitable to investigate all levels of microstructural features in advanced material systems.

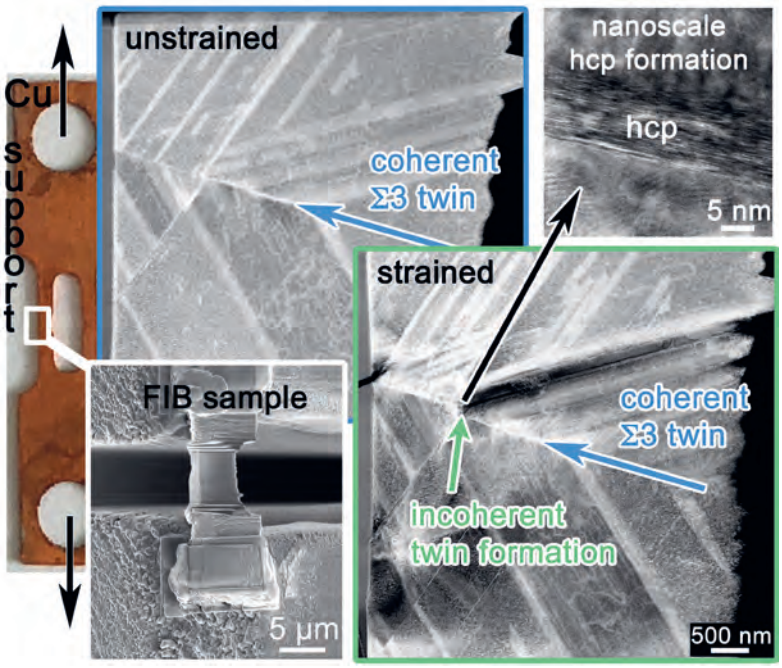


Fig. 1: *In situ TEM straining experiment of a dual phase high entropy alloy. The focused ion beam (FIB) prepared sample is welded onto the copper (Cu) support. During straining, an incoherent twin boundary segment is formed by the accumulation of dislocations at the coherent twin. This location serves as the nucleation point for the formation of the hexagonal closed packed (hcp) phase.*

In situ straining in the TEM in conjunction with novel imaging techniques provides unprecedented insights into the local deformation mechanisms of complex microstructures. As an example, the deformation induced formation of hexagonal close packed (hcp) phases at a coherent Σ3 twin boundary in the face centred cubic (fcc) grains of a dual phase high entropy alloy (HEA) is illustrated in Fig. 1. These novel alloy systems provide a unique combination of strength and ductility and *in situ* straining experiments are able to unlock the fundamental deformation induced phase transformation mechanisms [5]. *In situ* heating in the TEM [1] enables the determination of phase decomposition pathways and the local diffusion of elements on a nanometre level at temperatures of 1000 °C or more.

As a future perspective, *in situ* heating experiments will be coupled with electrical biasing to determine the effect of electric fields on the phase decomposition of materials and interfaces at atomic resolution. Also, combined *in situ* heating and mechanical loading experiments were developed over the last years and are now ready to investigate fundamental properties (e.g. activation energies for dislocation slip transfer) of plasticity, fracture and fatigue. The ultimate goal of the development of *in situ* electron microscopy techniques is to establish the underlying mechanisms linking microstructure and properties.

References

1. Harzer, T.P.; Duarte, M. J.; Dehm, G.: J Alloys Compd 695 (2017) 1583.
2. Betzler, S.B.; Harzer, T.; Ciston, J.; Dahmen, U.; Dehm, G.; Scheu, C.: Cryst Growth Des 16 (2016) 4309.
3. Folger, A.; Ebbinghaus, P.; Erbe, A.; Scheu, C.: ACS Appl Mater Interfaces 9 (2017) 13471.
4. Hieke, S.; Willinger, M.-C.; Wang, Z.-J.; Richter, G.; Chatain, D.; Dehm, G.; Scheu, C.: Acta Mater 165 (2019) 153.
5. Lu, W.; Liebscher, C.H.; Dehm, G.; Raabe, D.; Li, Z.: Adv Mater 30 (2018) 1804727.
6. Hodnik, N.; Dehm, G.; Mayrhofer K.J.J.: Acc Chem Res 49 (2015) 2022.

Submicron 3D Insights into Deforming Microstructures

C. Kirchlechner [1], J.-B. Molin [1], L. Renversade [1,2], J.-S. Micha [2]

[1] Department of Structure and Nano-/ Micromechanics of Materials (SN)
[2] CRG-IF BM32 at ESRF, Grenoble, France

Interlinking the mechanical properties of advanced materials to their underlying and evolving defect structure is key to understand and improve their reliability during daily use. For instance, in case of fatigue the properties of interest are the local strain distribution, the density and type of dislocations, crystal orientations and the crystallographic phase. All aforementioned properties are accessible at the sample surface with advanced characterization tools like scanning electron microscopy (SEM) including electron backscatter diffraction (EBSD) and, destructively, by transmission electron microscopy (TEM). But can we measure these properties *in situ*, non-destructively, with submicron precision and in bulk materials?

Within the XMicroFatigue project jointly funded by the German DFG and the French ANR, we are developing a unique tool being able to shed unprecedented light onto buried deforming volumes and interfaces. Together with our French partners from CEA-Grenoble we combined a synchrotron-based Differential Aperture X-ray Microscope (DAXM) [1] with a state-of-the-art micro-deformation rig [2]. The combined apparatus is located at BM32 of the European Synchrotron Radiation Facility (ESRF) and allows for *in situ* Laue microdiffraction experiments as well as a three dimensional reconstruction of the deformed sample volume. A spatial resolution better than 500 nm³ and deviatoric strain sensitivity better than 10^{-4} are currently reached.

The first project with this unique tool focusses on the fatigue damage initiation close to grain boundaries in copper. For this purpose micro cantilevers with a diameter of 10 µm are cut by focussed ion beam milling and subsequently cyclically loaded. Laue patterns and the force-displacement response are recorded during loading [3]. In addition, after ¼, ½, ¾ and after a full cycle the three dimensional dislocation structures and strain distributions are mapped with submicron resolution (Fig. 1).

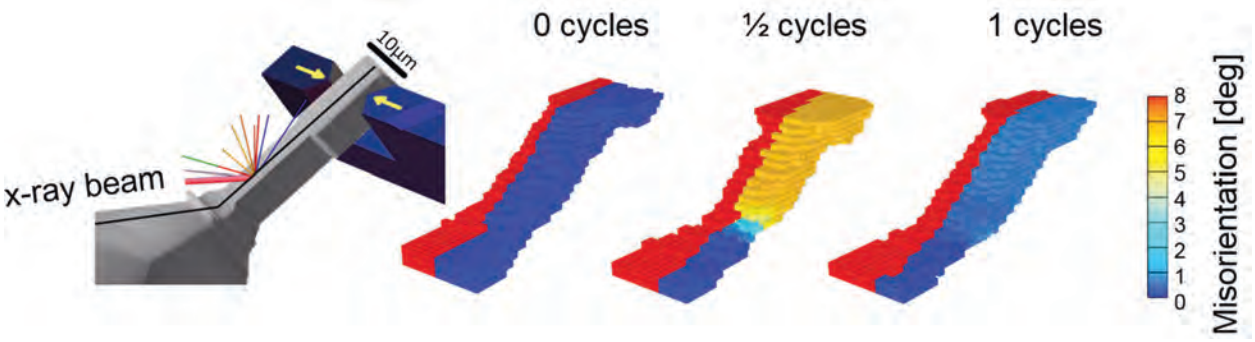


Fig. 1: *Bi-crystalline micro fatigue sample. The schematic indicates the location of the grain boundary (solid black line), the loading direction (yellow arrows) and the incoming and diffracted X-ray beams. The 3D point-to base disorientation reconstructions of the initial state, after ½ and a full cycle are plotted.*

References

1. Larson, B.C.; Yang, W.; Ice, G.E.; Budai, J.D.; Tischler, J.Z.: Nature 415 (2002) 887.
2. Kirchlechner, C.; Keckes, J.; Micha, J.S.; Dehm, G.: Adv Eng Mater 13 (2011) 837.
3. Kirchlechner, C.; Imrich, P.J.; Liegl, W.; Pörnbacher, J.; Micha, J.S.; Ulrich, O.; Motz, C.: Acta Mater 94 (2015) 69.

Environmental Small Scale Mechanics

S. Brinckmann, J. Duarte, V. G. Arigela, C. Kirchlechner

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

In the past decade, micromechanics have been established to fundamentally understand plasticity and fracture mechanisms and attribute these mechanisms to the microstructural components. At the MPIE, we develop analytical tools to investigate further the influence of environments on the plasticity and fracture mechanisms. High temperature applications in the energy sector require a thorough understanding of failure of microstructural components. Hydrogen is long known as detrimental for steels although the mechanisms at the microstructural length-scale are still not well understood.

Based on a previously build setup [1] for synchrotron experiments, a customized high-temperature microscale loading rig was developed to characterize material systems at their service conditions. This setup (Fig. 1) is being operated in a custom-made steel environmental chamber capable of attaining a vacuum of 10^{-6} mbar at temperatures of 600°C. The device also allows testing materials in various gas atmospheres permitting comprehensive understanding of environmental embrittlement phenomena. Moreover, this setup allows microscale mechanical testing using varying test methods (tension, compression, bending and fatigue), geometries, material systems, temperatures and environments.

Nanoindentation is a valuable technique to study hydrogen-microstructure interactions in metallic alloys, due to the small-probed volume and fast sample preparation. *In situ* testing while charging the sample with hydrogen prevents concentration gradients due to hydrogen desorption. Two custom electrochemical cells were built in-house to hydrogen-charge the samples during nanoindentation: "front-side charging" with the sample and the indenter tip immersed in the electrolyte, and "back-side charging" where the analyzed region is outside of the solution (Fig. 2). During front-side charging, surface degradation might occur which possibly reduces the reliability of post deformation analysis. Hence, we tailor the electrolyte and the operating procedures to the material and mechanical loading conditions to minimize the surface alteration and maximize the experimental stability.

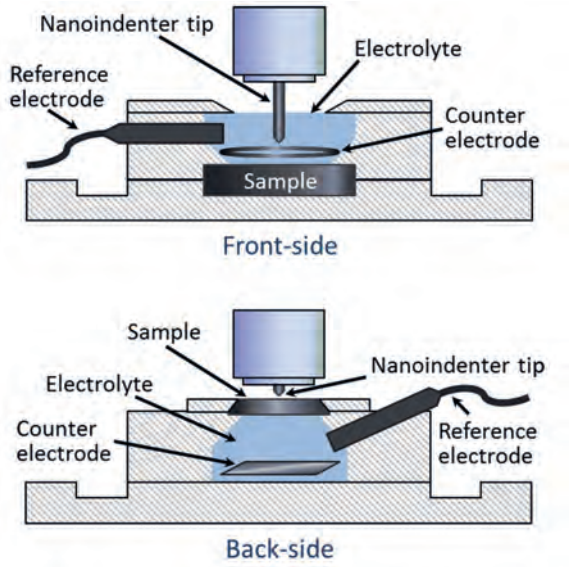


Fig. 2: *Schematic of the in-house electrochemical cells to perform in-situ nanoindentation.*

References

1. Kirchlechner, C.; Keckes, J.; Micha, J.S.; Dehm, G.: Adv Eng Mater 13 (2011) 837.

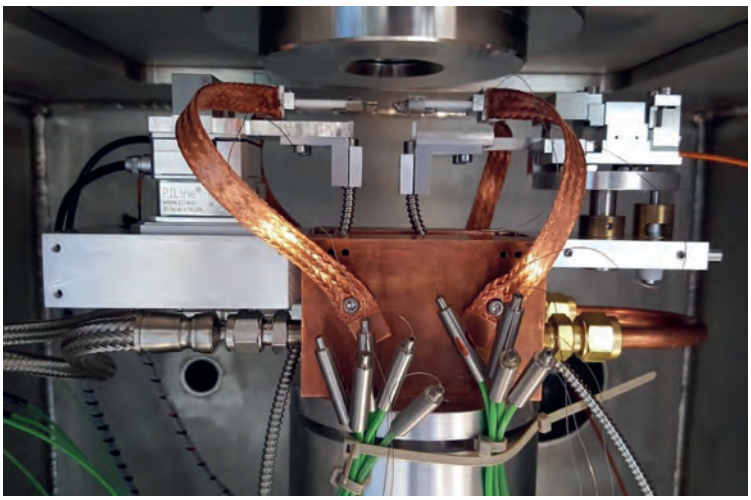


Fig. 1: *The high-temperature straining device enclosed in an environmental chamber.*

Micromechanics along Concentration Profiles in Diffusion Couples

F. Stein, W. Luo, C. Kirchlechner, S. Brinckmann

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

Mechanical properties of single-phase materials such as solid solutions or intermetallic phases may strongly vary as a function of composition (e.g. solid solution hardening by alloying), but are also well known to be affected by microstructural features such as size and crystallographic orientation of the grains and their morphology. Systematic investigations on the isolated, sole effect of composition often are difficult as for each composition a new alloy has to be prepared having not only a comparable microstructure but also keeping other parameters unchanged such as the level of impurities and the chemical homogeneity. Especially for intermetallic phases, an additional problem is the difficulty to produce flawless macroscopic samples, which are required for classical mechanical compression or bending tests.

To avoid these problems and to circumvent the necessity of preparing large-scaled samples, we follow a completely different approach, in which we use diffusion couples between two single-phase metals or intermetallics. By intimately contacting two pieces of such single-phase materials and holding this arrangement for sufficient times at elevated temperature, extended diffusion zones (in the order of 10 µm to 1 mm) of solid solutions and intermediate phases can develop having concentration gradients that cover the entire homogeneity range of these phases (for an example, see Fig. 1a). Under ideal conditions, large grains with a continuous concentration gradient grow in such diffusion zones.

Position-resolved nanoindentation tests along the concentration gradient (Fig. 1b) then are a simple method to yield information on the composition dependence of hardness and elastic modulus. Furthermore, by FIB (focused ion beam) cutting of small micropillars and microcantilevers at different selected positions in this diffusion zone parallel to the direction of the concentration gradient (Fig. 1c), we can obtain series of single-phase, single crystalline specimens with identical crystallographic orientation but different chemical composition. Then micropillar compression and microcantilever bending tests of these specimens allow a systematic study of composition- and orientation-dependence of strength and fracture toughness, for instance in order to get a better understanding of how the mechanical behaviour is affected by deviations from the ideal, stoichiometric composition of a phase, e.g., by introducing defects such as anti-site atoms in the crystal structure.

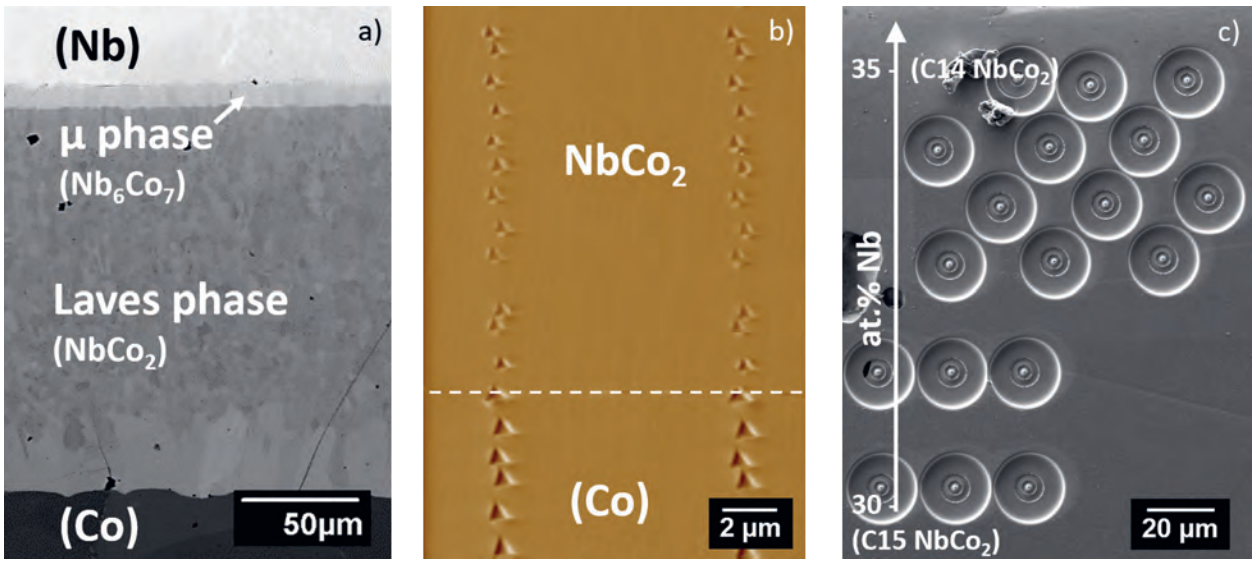


Fig. 1: *a) Diffusion couple of pure Co and Nb forming the (Co) and (Nb) solid solutions and two intermetallic phases, which are the Laves phase $NbCo_2$ covering a wide composition range from about 24 to 36 at.% Nb and the µ phase Nb_6Co_7 (47 to 55 at.% Nb); b) nanoindents along the concentration gradient near the boundary between (Co) and the Laves phase; and c) series of micropillars cut by FIB along and perpendicular to the concentration gradient in the diffusion zone of the Laves phase (showing the composition range where the structure type of the Laves phase changes from the cubic C15 to the hexagonal C14 variant).*

Advanced Metallurgical Synthesis and Processing

H. Springer, E. A. Jägle

Department of Microstructure Physics and Alloy Design (MA)

The metallurgical synthesis and processing is one of the cornerstones of the Institute, connecting therory and characterisation into the full circle of material development. It represents the basis to investigate micro- and nano-structural phenomena, validate simulations and study complex phase transformations and interfacial reactions. Accordingly a wide range of experiments are performed ranging from single-crystaline model alloys to modified industrial materials. About 500 metallic materials per year are produced for inhouse use across all departments as well as within the scope of external cooperations with scientific and industrial partners. Building on a strong set of basic equipment, the techniques and processes are constantly updated in parallel with the progress in materials development. With ever growing complexity, these innovative alloys are critically dependent on being developed together with their production techniques. At the same time, novel techniques require adapted alloy solutions, as they are opening the path for new concepts and material solutions.

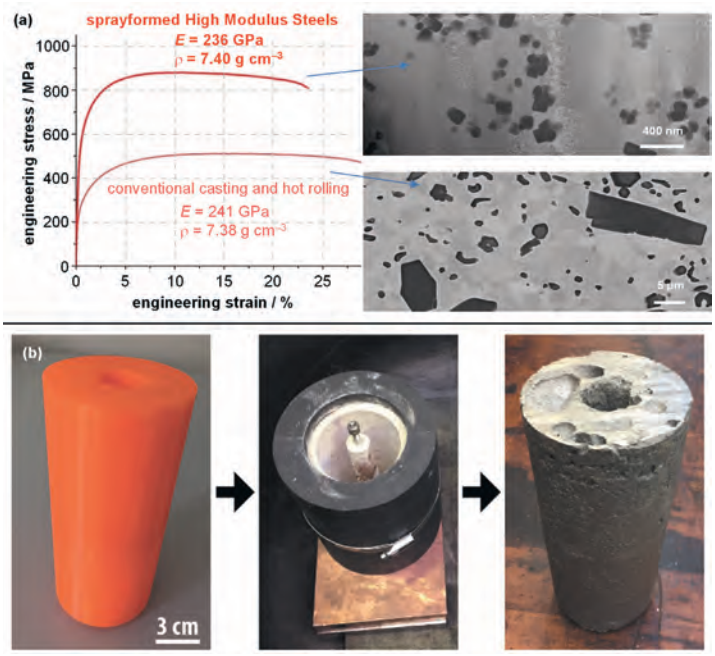


Fig. 1: *(a) Nano-structured High Modulus Steels obtained through sprayforming allow for an unprecedented combination of the key material properties for lightweight design. (b) Expendable 3D printed PLA polymer form being used to shape a sand casting mould, together with the final cast near-net shape ingot.*

Additive manufacturing, also referred to as 3D printing, is a prime example where the specific features of a novel technique both represent a challenge for utilising their full potential, as well as offering the possibility to realise materials with property combinations impossible to achieve before. The rapid solidification of the extremely small meltpool – compared to conventional large-scale casting procedures – can be used to achieve supersaturation of alloying elements, which then can be exploited for finely tuned precipitation reactions e.g. in Al-Sc alloys and maraging steels [1,2]. Another interesting pathway lies in utilising the increased solidification kinetics to obtain high modulus steels, which due to the *in situ* formation of nano-scaled TiB_2 particles, can overcome the inherent conflicts between density, stiffness, strength and ductility (Fig. 1a), and are therefore prime candidates for future lightweight design solutions [3]. By controlling the interaction between liquid powder particles and gases from the process atmosphere, oxides and nitrides can be synthesised in novel materials for high temperature applications [4]. Furthermore, additive manufacturing techniques can be used complementing the combinatorial high throughput design of structural materials [5], and as a rapid solidification technique which produces bulk samples rather than thin films or foils. 3D printing can also be used to produce expendable polymer shapes for pre-forming casting moulds (Fig. 1b; “lost-PLA casting”).

To fullfill the twin role as both the basis for material development, as well as the driving force for novel material design in highly innovative and competitive fields, new machinery (e.g. a strip casting simulator) are built and new labs are established at the MPIE, in close cooperations between groups inhouse and with external partners.

References

1. Jägle, E.A.; Sheng, Z.; Lu, L.; Wu, L.; Risse, J; Weisheit, A.; Raabe, D.: JOM 68 (2016) 943.
2. Kürnsteiner, P.; Wilms, M.B.; Weisheit, A.; Barriobero-Vila, P.; Jägle, E.A.; Raabe, D.: Acta Mater 129 (2017) 52.
3. Springer, H.; Szczepaniak, A.; Uhlenwinkel, V.; Raabe, D.: Sci Rep-UK 7 (2017) 2757.
4. Springer, H.; Baron, C.; Jägle, E.A.; Wilms, M.B.; Weisheit, A.; Raabe, D.: Mater Design 111 (2016) 60.
5. Knoll, H.; Ocylok, S.; Weisheit, A.; Springer, H.; Jägle, E.A.; Raabe, D.: Steel Res Int 87 (2016) 1.

Scanning Kelvin Probe Techniques for Advanced Measurement of Hydrogen and Electrochemical Activity at Buried Interfaces

M. Rohwerder, C.-H. Wu, W. Krieger

Department of Interface Chemistry and Surface Engineering (GO)

The utilization of Kelvin Probe (KP) techniques for spatially resolved high sensitivity measurement of hydrogen has been a major break-through for our work on hydrogen in materials [1]. A relatively straight forward approach was hydrogen mapping for supporting research on hydrogen embrittlement that was successfully applied on different materials, see e.g. [2-6].

The key strength of this KP approach for H mapping is that the hydrogen release from the investigated material into an ultra-thin palladium detection layer is directly measured with high local resolution. This release rate is a measure for how fast H can be supplied e.g. to a crack tip. Scanning KP (SKP) is fully quantitative. We are currently working on several concepts for full calibration also for Scanning Kelvin Probe Force Microscopy (for instance by a combination (see [5]).

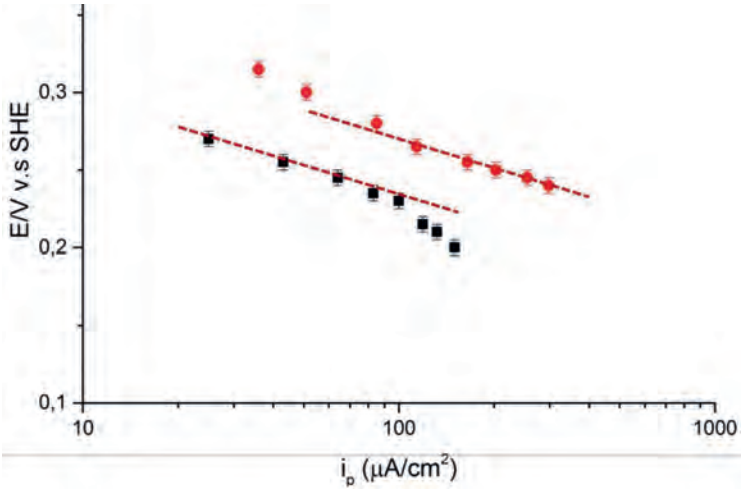


Fig. 1: *I(U) relationship between ORR and HOR on two iron(oxide) surfaces, one slightly (black) and one highly defective (red). Atmosphere O_2 at about 90% r.h., i.e. the Fe is covered by less than 1 nm of water.*

Another idea is to use the method for detecting corrosion sites through the metal. This is of interest, for instance, for fundamental studies in corrosion science as well as for practical application in the field, e.g. to measure internal corrosion at the outside of a pipeline.

Since often the evaporation of a thin Pd film and providing a dry nitrogen atmosphere is inconvenient or even impossible (such as in the field), alternatives are currently being investigated. Another problem is saturation of the potential change upon reaching certain levels of H activity. This can be circumvented by using another approach where the equilibrium potential between hydrogen oxidation (HOR) and oxygen reduction reactions (ORR) is used for measuring permeation/effusion rates of hydrogen, which was first applied for investigating oxygen reduction at buried interfaces (see [7,8] and p. 66). We found that this can even be applied directly on the iron (oxide) surface (see fig.1).

References

1. Evers, S.; Senöz, C.; Rohwerder, M.: Sci Technol of Adv Mat 14 (2013) 014201.
2. Koyama, M.; Rohwerder, M.; J Mater Sci Techol 33 (2017) 1481.
3. Nagashima, T.; Koyama, M.; Bashir, A.; Rohwerder, M.; Tasan, C.C.; Akiyama, E.; Takai, K.; Raabe, D.; Tsuzaki, K.: Mater Corros 68 (2017) 306.
4. Koyama, M.; Bashir, A.; Merzlikin, S.V.; Rohwerder, M.; Akiyama, E.; Tsuzaki, K.; Raabe, D.: J Electrochem Soc 162 (2015) C638.
5. Krieger, W.; Merzlikin, S.V.; Szczepaniak, A.; Springer, H.; Rohwerder, M.: Acta Mater 144 (2018) 235.
6. Tarzimoghadam, Z.; Rohwerder, M.; Merzlikin, S.V.; Bashir, A.; Yedra, L.; Eswara, S.; Ponge, D.; Raabe, D.: Acta Mater 109 (2016) 69.
7. Vijayshankar, D.; Tran, T.H.; Bashir, A.; Evers, S.; Rohwerder, M.: Electrochim Acta 189 (2016) 111.
8. Vijayshankar, D.; Altin, A.; Merola, C.; Bashir, A.; Heinen E.; Rohwerder, M.: J Electrochem Soc 163 (2016) C778.

Coupled Electrochemical Scanning Flow Cell-Based Techniques for Electrocatalysis Research

S. Cherevko [1], O. Kasian [1], S. Geiger [1], M. Ledendecker [1], K.J.J. Mayrhofer [1, 2]

[1] Department of Interface Chemistry and Surface Engineering (GO)
[2] Helmholtz Institute Erlangen-Nürnberg for Renewable Energy

Electrochemical scanning flow cell (SFC) has been developed in the "Electrocatalysis" group of the GO department to assist in the electrochemical measurements in research areas such as corrosion, fuel cells, water electrolysis, electrosynthesis, etc. Due to its high-throughput nature, this cell transformed typically manual, routine, tedious, and time consuming measurements into fast and automized ones. What made SFC especially powerful, however, is its scaling capability to create new, and unique for electrocatalysis research, coupled techniques such as *in situ* SFC inductively coupled plasma mass spectrometry (*in situ* SFC-ICP-MS), SFC on-line electrochemical mass spectrometry (SFC-OLEMS), and others (see Fig. 1). Recently, employing a set of SFC-based techniques, in this case SFC-ICP-MS and SFC-OLEMS, "Electrocatalysis" group aided in understanding of the water splitting mechanism on iridium,[1-3] a very important and timely topic in the renewable hydrogen production technology.

In the years that past, potential of these techniques has been realized by numerous research groups worldwide, which is supported by a growing number of the high quality scientific works employing *in situ* ICP-MS and similar methods. Both, internal and external developments reveal that, despite the fact that SFC can be considered as a mature technique, the full potential of this technique is still yet to be seen. Development is continuing in the direction of SFC coupling to Raman, infra-red, X-ray based spectroscopies, etc.

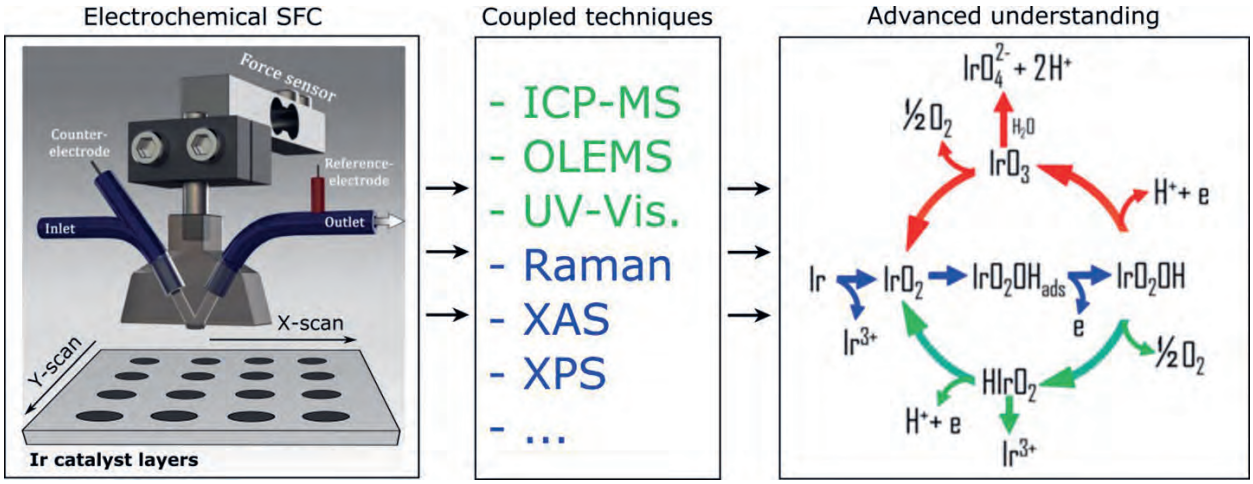


Fig. 1: *Concept of the combinatorial reaction analysis by SFC coupled techniques.*

References

1. Kasian, O.; Grote, J.; Geiger, S.; Cherevko, S.; Mayrhofer, K.J.J.: Angew Chem Int Ed Engl 57 (2018) 2488.
2. Li, T.; Kasian, O.; Cherevko, S.; Zhang, S.; Geiger, S.; Scheu, C.; Felfer, P.; Raabe, D.; Gault, B.; Mayrhofer, K.J.J.: Nat Catal 1 (2018) 300.
3. Geiger, S.; Kasian, O.; Ledendecker, M.; Pizzutilo, E.; Mingers, A.; Fu, W.; Diaz-Morales, O.; Li, Z.; Oellers, T.; Fruchter, L.; Ludwig, A.; Mayrhofer, K.J.J.; Koper, M.; Cherevko, S.: Nat Cat (2018) 508.

Ab initio Description of Electrified Interfaces in Electrochemistry and Atom Probe Tomography

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

[1] Department of Computational Materials Design (CM)
[2] Department of Interface Chemistry and Surface Engineering (GO)

Solid/liquid interfaces are an essential constituent in electrochemistry, electrocatalysis and corrosion. These interfaces are characterized by strong electric fields that originate from finite surface charges and modify adsorption, desorption, interface reconstructions, double layer formation and reactions at the electrode/electrolyte interface. Electric fields (a few 10^{10} V/m) are also required for field evaporation, the key mechanism in atom probe tomography (APT). Modelling such electrified interfaces is challenging: while the counter-charges in reality are far away, atomistic simulations must either include artificial ones or introduce special boundary conditions, which may cause additional complications. In consequence, no standard approach is yet available. The groups of C. Freysoldt, M. Todorova and S. Wippermann develop and employ various methods to treat finite electric fields in density-functional theory (DFT).

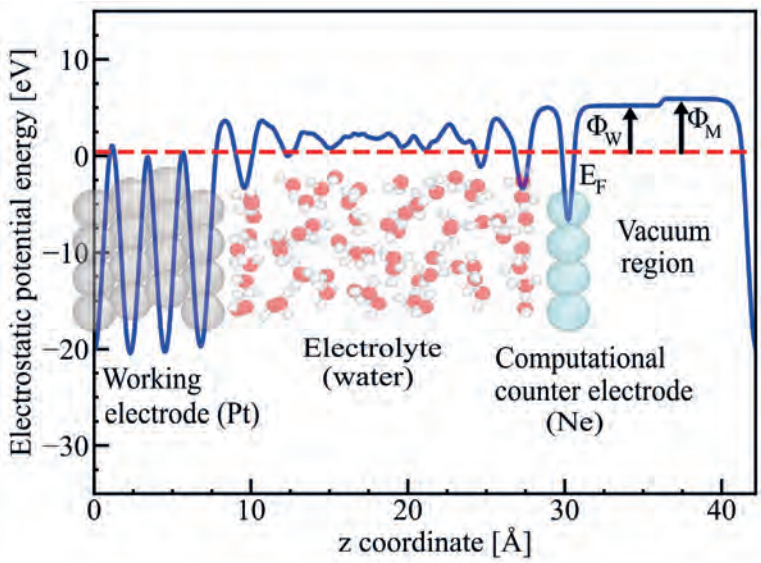


Fig. 1: *Supercell-setup of the computational counter electrode. Atoms are shown as spheres (grey: Pt, red: O, white: H, light blue: Ne). The blue line shows the electrostatic potential distribution within the supercell. The Fermi energy of Pt is shown as a red dashed line. The dipole correction, via which the potential of the interface is determined is visible as a jump in the electrostatic potential in the vacuum region, i.e. difference between the work function of the metal (Φ_M) and the work function of the counter electrode (Φ_W).*

Combining concepts from electrochemistry and semiconductor physics, we were able to design a novel approach that allows to study chemical reactions at the electrochemical interface using standard DFT codes [1]. The schematic setup of the approach is shown in Fig. 1 and described in detail on p. 193. An alternative approach is the modern theory of polarization, which introduces an electric field term directly into the DFT Hamiltonian. This allows us to simulate the full electrochemical cell without a vacuum region.

Studying elementary processes at APT tip surfaces by DFT, requires, in contrast, a voltage drop of a few 10 V across the simulation cell. This is much larger than the typical energy scale of valence electrons, precluding any attempts to generate the fields by charged ions. In this case, we model the electrified surface by a charged slab in a periodic cell. In all existing DFT codes the counter charge is smeared out, which produces an uncontrollable distribution of the electric fields. We therefore developed a new approach that places a counter electrode in the centre of the vacuum region together with a discontinuity in the electrostatic potential, a technique known as the “dipole correction” for asymmetric slabs [2]. This combination realizes a single “generalized dipole correction”, which enables, together with further improvements ensuring a proper initialization and suppression of charge fluctuations, surface calculations for fields even beyond the evaporation threshold.

Our development activities aim at solving outstanding questions related to electrified surfaces, and profit from mutual insights generated in the different areas of application. To foster exchange also at an international scale, in March 2017 we conducted a three-day workshop at Ringberg castle on “High Electric Fields in Electrochemistry and Atom Probe Tomography”, to bring together leading scientists from the areas of APT, field-ion microscopy and electrochemistry.

Time scales of electrochemical processes are far beyond the realm of DFT. Future work will in addition explore how to combine the electric field approaches with accelerated sampling techniques or implicit solvent approaches.

References

1. Surendralal, S.; Todorova, M.; Finnis, M.; Neugebauer, J.: Phys Rev Lett 120 (2018) 246801.
2. Freysoldt, C.; Neugebauer, J.: Phys Rev B 97 (2018) 205425.

Ab initio Thermodynamics and Long-Term Archiving with the in-house Developed *pyiron* Platform

J. Janssen, B. Grabowski, T. Hickel, J. Neugebauer

Department of Computational Materials Design (CM)

A long-term project in the CM department is the development of a Python based platform, called pyiron [1], to handle complex simulation protocols together with the huge amount of generated ab initio and atomistic data. In the following, we discuss two major applications.

Ab initio thermodynamics: The CM department is systematically developing and exploring *ab initio* based approaches to describe materials properties over the entire temperature range up to and even beyond melting. This requires novel strategies to compute all relevant entropic contributions, as well as their non-adiabatic coupling phenomena. The systematic achievement of highest precision and efficiency requires combining first principles calculations performed with different codes with advanced statistical sampling methods. One of the strategies – the local anharmonicity (LA) approach – explores, for example, the leading terms of fully anharmonic lattice vibrations. Another strategy (TU-TILD) uses on-the-fly optimized potentials to sample the configuration space. A third one accurately describes paramagnetic disorder using spin-space averaging (SSA).

Therefore, complex simulation protocols that combine distinctly different computer codes and that handle hundreds or thousands of separate calculations have to run on heterogeneous computer architectures. To enable the development and dissemination of these complex simulation protocols, the CM department has developed *pyiron* [1]. Like an integrated development environment (IDE) for programming languages, *pyiron* allows one to interactively implement and test simulation protocols and to upscale them for high-throughput simulations on large computer clusters. The key idea behind this new framework is to provide a single tool with a unified interface for all routines developed and established in the department or externally. This enables the user to focus fully on science rather than having to deal with technicalities and specific input/output formats of the various codes and tools.

While the development of a Python based workbench started back in 2011, the recent developments towards abstraction of the underlying concepts resulted in the release under an open source license in 2018 (www.pyiron.org). This allows the CM department to make newly developed approaches and computational tools accessible to a broad community. Successful examples for the application of *pyiron* include the simulation of the solid-liquid transitions with the two-optimized references thermodynamic integration (TOR-TILD) [2], paramagnetic defect calculations with spin-constraints, *ab initio* calculations under controlled potentiostat conditions for electrochemical systems [3], as well as machine-learning techniques for the calculation of interstitial solution enthalpies. With this versatility, the *pyiron* framework has become more and more established in the CM department. It has further been used in several collaborating institutes, e.g., for a high-throughput framework to validate empirical potentials developed at the Interdisciplinary Centre for Advanced Materials Simulation, ICAMS.

Fig. 1: *By using the integrated development environment pyiron, the modelling strategies become easily readable in interactive notebooks (middle). They provide a direct link to the physical concepts and raw data (left) as well as to the final results appearing in publication (right).*

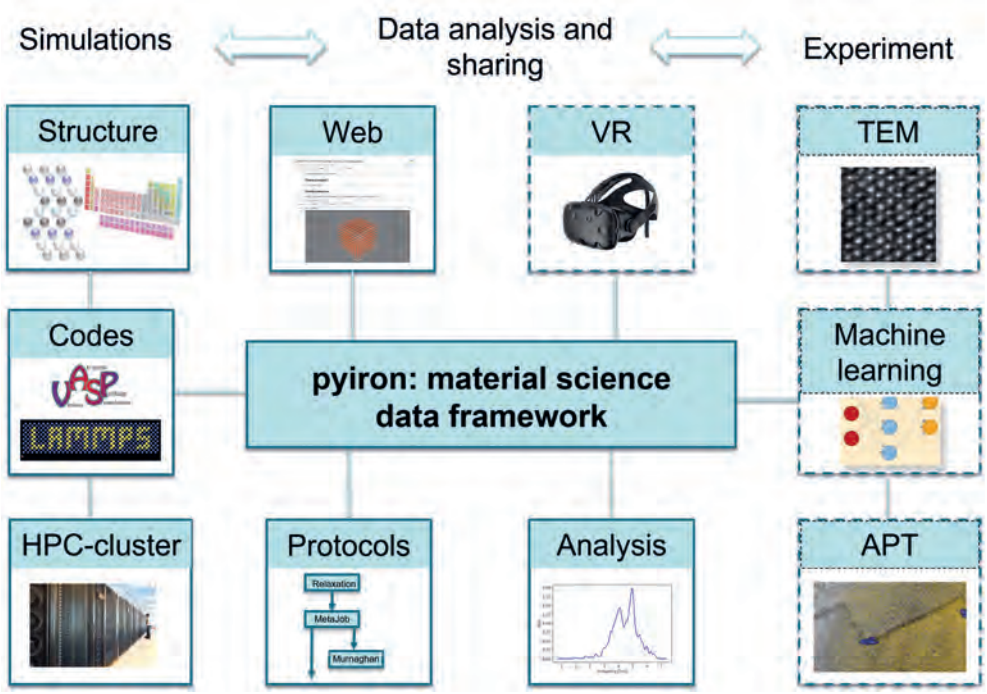


Fig. 2: *Overview of the pyiron framework covering all aspects of data generation, collection, storage, sharing, and analysis. While originally developed to support the simulation activities of the CM department it evolved into a versatile tool that can also handle experimental data. The dashed lines mark topics and software modules under development. (VR: Virtual Reality; HPC: High-Performance Computing; APT: Atom Probe Tomography)*

Long-term archiving: The last two years showed that the algorithmic concepts and solutions to realize *pyiron* allow applications that are much broader than the original goal of developing and working with complex simulation protocols (see Fig. 2). For example, the fundamental concept of *pyiron* objects allowed to link a computationally highly efficient but complex storage solution combining SQL with the HDF5 file format and a conventional file system with an easy to learn and unified user interface. The experience with such a new hierarchical storage solution based on Python, which reflects the needs of high-throughput simulations with various computer codes, turns out to be easily adaptable to store and process not only the huge datasets generated by the advanced in-house experimental tools but also the complex experimental workflows together with their respective metadata (as explored in BigMax, p.53). Examples are high-resolution TEM (transmission electron microscopy) pictures or atom-probe tomography micrographs.

A major advantage of having a unified platform is that it not only enables an easy transfer of data from simulation to experiment but allows vice versa an easy transfer of knowledge and software tools between the experimental and theoretical activities. Machine learning tools enabled converting a few thousand pictures measured by field ion microscopy (FIM) into a 3D atomistic model [4]. This model was then fed into an atomistic MD (molecular dynamic) simulation to obtain a fully relaxed structure that removes experimental noise and systematic imaging errors. The ensuing high quality data allow one to detect even fine details such as point defects (vacancies) in the sample. Linking data and tools, and providing all details in interactive and ready to use jupyter notebooks, makes *pyiron* a highly productive tool: It allows researchers to handle all the data-related requirements encountered in advanced correlative materials science research projects that require the use of a multitude of experimental and theoretical concepts and tools.

An additional benefit of *pyiron* is its ability to log all the steps of the user in constructing the protocols and achieving the results. Thus, *pyiron* not only stores all the input and output data but automatically also all the metadata related to scientific setup and workflow, such as, e.g., the program and code versions that have been used, the previous calculations/ measurements that have been employed as input or the type of analysis or data conversion that has been implemented. With the ongoing developments, we expect pyiron to become the major tool at the MPIE for an active management of big data in modelling and experiment.

References

1. Janssen, J.; Surendralal, S.; Lysogorskiy, Y.; Todorova, M.; Hickel, T.; Drautz, R.; Neugebauer, J.: Comp Mater Sci (2018) – in print.
2. Zhu, I.; Grabowski, B.; Neugebauer, J.: Phys Rev B 96 (2017) 224202.
3. Surendralal, S.; Todorova, M.; Finnis, M.W.; Neugebauer, J.: Phys Rev Lett 120 (2018) 246801.
4. Katnagallu, S.; Gault, B.; Grabowski, B.; Neugebauer, J., Raabe, D.; Nematollahi, A.: Mater Charac 146 (2018) 307.

The Düsseldorf Advanced Material Simulation Kit: DAMASK

F. Roters [1], P. Shanthraj [1,2], M. Diehl [1], P. Eisenlohr [1,3], D. Raabe [1]

[1] Department of Microstructure Physics and Alloy Design (MA)
[2] now at: School of Materials, University of Manchester, UK
[3] now at: Chemical Engineering & Materials Science, Michigan State University, USA

Crystal plasticity modelling has gained considerable momentum in the past 20 years [1]. Developing this field from its original mean-field homogenization approach in conjunction with viscoplastic constitutive hardening rules into an advanced multi-physics 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 packages Abaqus and MSC.Marc were developed.

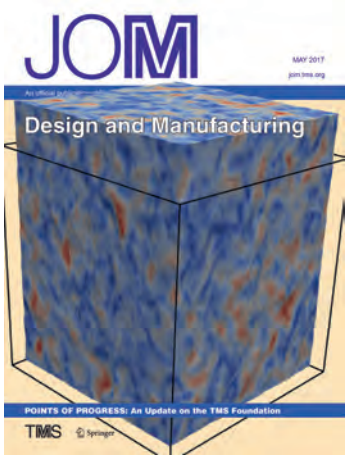


Fig. 1: *Cover of May 2017 JOM issue featuring DAMASK RVE.*

The scientific focus of the group is, 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" (CMC^n, the first joined project between the Max Planck and the Fraunhofer Society 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 2010, in collaboration with Prof. R. Lebensohn (LANL, Humboldt awardee at the MPIE at that time), a spectral method based boundary value problem solver, especially suited for RVE simulations (Fig. 1), was added to complement the commercial FEM solvers. In September 2011, a website (https://damask.mpie.de) was launched to release the code to the public domain as free software according to GPL 3. The idea of a flexible open source CP implementation was very well received by the scientific community. Presently, more than 50 groups across the world use DAMASK [4], including universities such as University of California Los Angeles (UCLA), research facilities such as LANL, and multinational companies such as Tata Steel. These groups contribute to the further code development as well, e.g. by adding features such as new or modified constitutive models [5]. In 2015, multi-physics extensions were incorporated into DAMASK to consistently treat coupled problems, such as thermo-mechanics, chemo-mechanics [6], and damage-mechanics [7]. Since early 2016, the code is hosted in a public repository using GitLab (https://magit1.mpie.de) to enable and assist collaborative development among the growing user community.

References

1. Roters, F.; Eisenlohr, P.; Hantcherli, L.; Tjahjanto, D.D.; Bieler, T.R.; Raabe, D.: Acta Mater 58 (2010) 1152.
2. Kalidindi, S.R.; Bronkhorst, C.A.; Anand, L.: J Mech Phys Solids 40 (1992) 537.
3. Ma, A.; Roters, F.: Acta Mater 52 (2004) 3603.
4. Roters, F.; Diehl, M.; Shanthraj, P.; Eisenlohr, P.; Reuber, C.; Wong, S.L.; Maiti, T.; Ebrahimi, A.; Hochrainer, T.; Fabritius, H.O.; Nikolov, S.; Friak, M.; Fujita, N.; Grilli, N.; Janssens, K.G.F.; Jia, N.; Kok, P.J.J.; Ma, D.; Meier, F.; Werner, E.; Stricker, M.; Weygand, D.; Raabe, D.: Com Mater Sci (2019).
5. Cereceda, D.; Diehl, M.; Roters, F.; Shanthraj, P.; Raabe, D.; Perlado, J.M.; Marian, J.: GAMM-Mitt 38 (2015) 213.
6. Svendsen, B.; Shanthraj, P.; Raabe, D.: J Mech Phys Solids 112 (2018) 619.
7. Shanthraj, P.; Sharma, L.; Svendsen, B.; Roters, F.; Raabe, D.: Comput Method Appl M 312 (2016) 167.

Large-scale and Networking Projects

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

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

[1] Research Coordination Office
[2] Department of Interface Chemistry and Surface Engineering (GO)

The IMPRS-SurMat is an established and highly visible doctoral programme in the field of materials science in North Rhine-Westphalia, existing for 15 years and attracting highly qualified students from all over the world. It plays an essential role in gaining and promoting young scientists for all partner institutes of the school.

As a structured, three-year doctoral programme, the IMPRS-SurMat is coordinated at the Max-Planck-Institut für Eisenforschung (MPIE). Currently, it is in the middle of the third funding period from January 2016 until December 2021. Each running period lasts 6 years. The SurMat was successfully evaluated twice, in 2009 and 2014. The school is open for students with a Master's degree in Materials Science, Physics, Chemistry, or related subjects and offers funding for 3 years.

In 2004, the SurMat was founded by the MPIE, the Max-Planck-Institut für Kohlenforschung in Mülheim and the Ruhr-Universität Bochum. Two new partner institutes joined the SurMat in 2015: the Max-Planck-Institut for Chemical Energy Conversion and the University Duisburg-Essen. Therefore, today the main players of the Rhine-Ruhr region in the fields of materials science and interfaces are interlinked within the IMPRS-SurMat.

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

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

The various PhD projects are interdisciplinary with at least two advisors from different academic departments. In addition, the close link between experimental and computational science leads many projects to outstanding results.

Besides the regular funding of the Max Planck Society and the involved partner institutes the SurMat gained additional funding for the third running period from the state North Rhine-Westphalia and from Tata Steel,

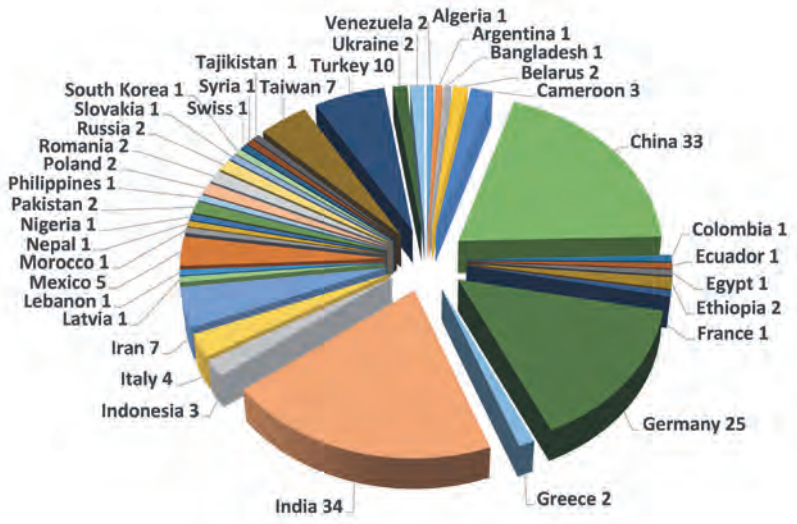


Fig. 1: *Origin of IMPRS-SurMat students by countries.*

Fig. 2: *IMPRS-SurMat community at the annual retreat in 2018.*

The Netherlands, that supports two projects of SurMat students.

During the first half of the current running period, the new partner institutes integrated very well in the school. Scientists of the University Duisburg-Essen as well as of the MPI for Chemical Energy Conversion started to contribute to SurMat lectures and several talented students started very promising research projects at both institutes. Research at the University Duisburg-Essen focuses amongst others on nanoparticles. The MPI for Chemical Energy Conversion concentrates on Carbon-based materials, e.g. designed for electrochemical applications.

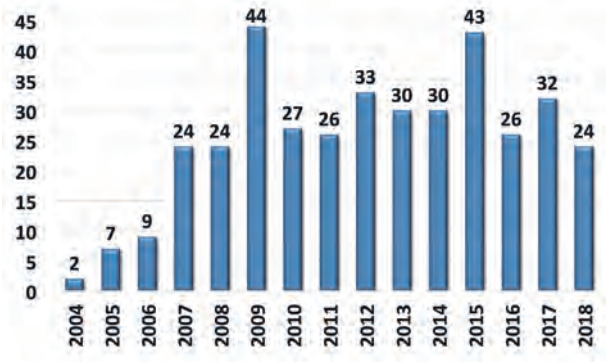


Fig. 3: *Number of publications by IMPRS-SurMat students in peer reviewed journals.*

The SurMat hosted already two workshops in the third running period: in fall 2016 the "Materials Day" showcased recent research highlights from the Materials Research Department of the Ruhr-Universität Bochum and the IMPRS-SurMat. The first topical joint Workshop of the IMPRS-SurMat and the IMPRS-RECHARGE "Towards an Atomistic Understanding of Reactions at Surfaces and Interfaces" was conducted at the MPIE in fall 2018. The IMPRS-RECHARGE (International Max Planck Research School on Reactive Structure Analysis for Chemical Reactions) started in 2015 at the MPI for Chemical Energy Conversion. The purpose of this joint workshop was to discuss recent developments, methods and tools to reveal reaction mechanisms and elemental steps at the atomistic scale in catalysis, electrochemistry and corrosion and to identify possible common activities.

In addition, these workshops provided an excellent opportunity for an active exchange of research results and ideas between doctoral students and advisors. Thus, they also enhance the cooperation between the SurMat partner institutes.

For the doctoral students, the school offers 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 core curriculum remains to be delivered over years one and two. Until 2018, there was a series of four two-week classes in each term (winter/summer). The SurMat board decided to restructure these classes into five two-day lectures starting from 2019 to focus strongly on the main topics in modern materials and interface science. Furthermore, welcome days for new SurMat students shall be established in 2019. SurMat offers soft skills trainings such as presentation skills, scientific writing and project management, as well as German language courses and career development.

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.

Since 2004, 167 students from 37 different countries joined the IMPRS-SurMat. While the majority came from Asia (China, India, Iran), several students originated from Europe, Africa, Middle and South America. For admission, the students have to apply online. Either one or two application rounds are conducted per year. The application process has continuously evolved into an efficient selection procedure. All shortlisted applicants are invited for personal interviews. For the third running period already 61 new students could be won (2015-2018), which means a significant growth of the SurMat community.

Also since 2004, 89 students have successfully finished their doctorate within the IMPRS-SurMat, 19 of them with distinction. The doctoral work requires on average between 3 and 4 years with a genuinely high scientific output. 381 journal articles were published, as well as more than 260 poster and 270 oral presentations on conferences.

BigMax: Max Planck Research Network on Big-Data-Driven Materials Science

Z. Peng [1], M. Ashton [2], D. Raabe [1], B. Gault [1], J. Neugebauer [2], C. Freysoldt [2]

[1] Department of Microstructure Physics and Alloy Design (MA)
[2] Department of Computational Materials Design (CM)

Materials science is data-intensive, with nowadays huge data acquisition rates in a wide array of experimental and computational techniques. Dealing with vast amounts of data is not just a technical challenge, it is an opportunity for new discoveries. A large-scale collaborative MaxNet on Big-Data-Driven Materials Science, i.e. BigMax, blending materials scientists and data scientists was hence recently established. Information extraction from atom probe tomography (APT) is a new playground for machine-learning.

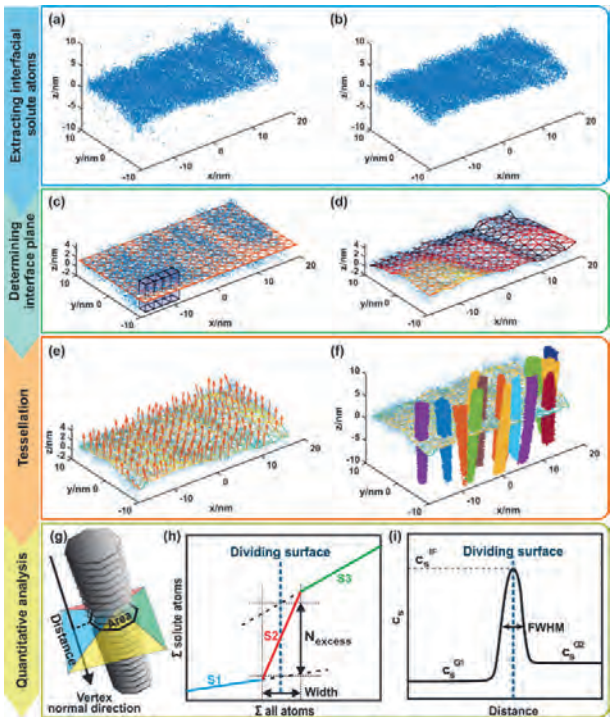


Fig. 1: *Interfacial solutes (a) are extracted through DBSCAN (b), PCA is applied to set up the initial interface plane, (c), based on which, the final interface is determined (d). In the third step, the normal direction of each vertex is calculated (e), and the dataset is split in sub-volumes (f) from which profiles are extracted and processed [1].*

Upon processing and reconstruction, APT data is a three-dimensional point cloud from which precise compositional information needs to be extracted. Interphase interfaces and grain boundaries are crucial microstructural features, which generally adopt complex three-dimensional shapes. Therefore obtaining a complete view of the distribution of atomic species near interfaces can be extremely challenging. Two typical machine-learning techniques, a method for finding clusters in point clouds, i.e. DBSCAN, as well as principal component analysis (PCA) were used to find the position of an interface and derive its local orientation. After PCA and DBSCAN, profiles are calculated along the local normal to the surface from which the composition, interfacial width and Gibbsian interfacial excess can be derived and plotted in the form of a two-dimensional map, as shown in Fig. 1.

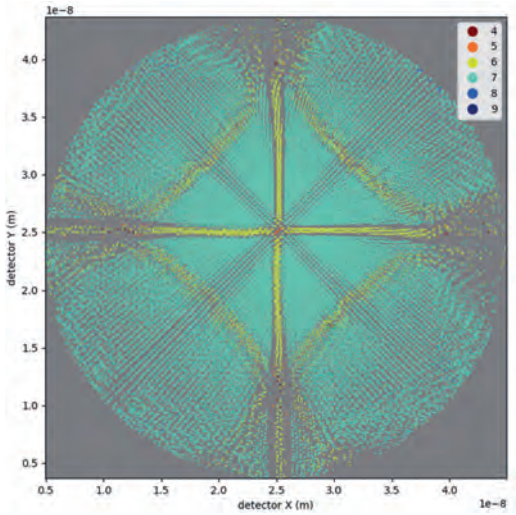


Fig. 2: *Detected positions of approximately 50,000 evaporation events for a [110]-oriented tungsten needle simulated in TAPSim. Each detected event is coloured according to the atom's coordination number immediately prior to its evaporation.*

On the other hand, the positions of hundreds of millions of atoms may contain hidden patterns and features relating to material properties. Within the BigMax network, we therefore collaborate with the Fritz-Haber-Institute on establishing structure-property relationships from APT data. In parallel, we pursue novel ideas for improving the reconstruction of the 3D atomic positions from the APT raw data. New algorithms are being developed and trained with APT forward simulations. For instance, Fig. 2 shows a simulated detector hit map, revealing a clear correlation between focussing effects in the pole structure and coordination within the surface. The simulations are augmented by systematic theoretical investigations of the field-evaporation mechanism at the atomic scale.

References

1. Peng, Z.; Lu, Y.; Hatzoglou, C.; Kwiatkowski da Silva, A.; Vurpillot, F.; Raabe, D.; Gault, B.: Microsc Microanal, under review.

Networking within MAXNET Energy Consortium

O. Kasian [1], S. Cherevko [1], E. Pizzutilo [1], A. Sarfraz [1], A. Erbe [1,2], K.J.J. Mayrhofer [1,3]

[1] Department of Interface Chemistry and Surface Engineering (GO)
[2] Norwegian University of Science and Technology
[3] Helmholtz Institute Erlangen-Nürnberg for Renewable Energy

The intermittent nature of renewable energy from wind and solar requires the development of efficient solutions for conversion and storage on the TWh scale. The focus of the MAXNET Energy research cooperation are key reactions for the synthesis of storage molecules and added-value chemicals utilizing renewable energy. The span of the overall projects of the partners thereby ranges from atomistic insights of catalytic reactions to fundamentals of engineering solutions for technical systems [1].

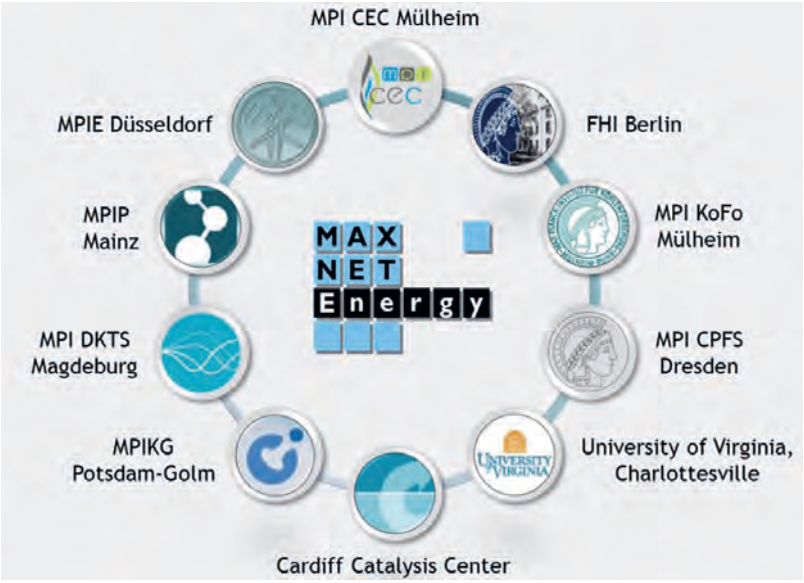


Fig. 1: *Partner institutes within the MAXNET Energy network.*

The activities of the GO department within the MAXNET Energy consortium are mainly related to fundamental studies of electrochemical reactions, the catalyst interface and its changes due to different operation conditions . The electrocatalysis group was additionally active in two particular projects in collaboration with the associated Cardiff Catalysis Institute (CCI) on the on-Site production of hydrogen peroxide with Au-Pd catalysts in electrocatalysis and heterogeneous catalysis and on the oxygen evolution reaction (OER) on Ir-Ru mixed oxide nanoparticles, as well as on Ir-modified TiO_2 nanotubes in collaboration with the associated University of Virgina. The CCI synthesized well-defined Au-Pd and Ir-Ru-based nanoparticles with different compositions, sizes and supports. The University of Virginia additionally prepared various TiO_2-nanotubes as model support materials, which were successfully modified with Ir using physical vapour deposition. Those catalysts were then transferred to the MPIE for their electrochemical and structural characterization. The electrocatalytic performance was fundamentally investigated using the rotating ring disc electrode (RRDE). The microstructural and compositional changes were instead characterized at different stages of the accelerated degradation protocols (SFC-ICP-MS) with identical location TEM (IL-TEM). The strong combination in synthesis of the particular interesting material classes by the partners within MAXNET Energy and the unique characterization techniques at the MPIE led to a comprehensive understanding of the relationship between the structure and activity/stability for the catalysts [2,3,4].

References

1. Auer, A.A.; Cap, S.; Antonietti, M.; Cherevko, S.; Deng, X.; Papakonstantinou, G.; Sundmacher, K.; Brüller, S.; Antonyshyn, I.; Dimitratos, N.; Davis, R.J.; Böhm, K.; Fechler, N.; Freakley, S.; Grin, Y.; Gunnoe, B.T.; Haj-Hariri, H.; Hutchings, G.; Liang, H.; Mayrhofer, K.J.J.; Müllen, K.; Neese, F.; Ranjan, C.; Sankar, M.; Schlögl, R.; Schüth, F.; Spanos, I.; Stratmann, M.; Tüysüz, H.; Vidakovic-Koch, T.; Yi, Y.; Zangari, G.: Green 5 (2015) 21.
2. Choi C.H.; Choi, W.S.; Kasian, O.; Mechler, A.K.; Sougrati, M.T.; Brüller, S.: Angew Chem-Ger Edit 129 (2017) 8935.
3. Pizzutilo, E.; Kasian, O.; Choi, C.H.; Cherevko, S.; Hutchings, G.J.; Mayrhofer, K.J.J.; Freakley S.: Chem Phys Let 683 (2017) 436.
4. Kasian, O.; Geiger, S.; Stock, P.; Polymeros, G.; Breitbach, B.; Savan, A.; Ludwig, A.; Cherevko, S.; Mayrhofer, K.J.: et al.; Electrochem Soc 163 (2016) F3099.

Fraunhofer – Max-Planck – Cooperation: Advanced Process and Alloy Design for Laser Additive Manufacturing of Metals (AProLAM)

E. A. Jägle [1], A. Weisheit [2]

[1] Department of Microstructure Physics and Alloy Design (MA)
[2] Fraunhofer Institute for Laser Technology ILT, Aachen, Germany

This collaborative research project was active from summer 2015 until the end of 2018. It aimed to combine the expertise of the two participating institutes in Additive Manufacturing (AM) process development and alloy design, respectively. The work focused on two research hypotheses:

(i) Due to the cyclic re-heating of material deposited track-by-track and layer-by-layer in AM, the so-called intrinsic heat treatment, it should be possible to design and fabricate a material that gets precipitation strengthened during the AM process.

(ii) Due to the strong melt pool convection (Marangoni effect) and high cooling and solidification rates during AM, the production of Oxide Dispersion Strengthened (ODS) steels without laborious and costly mechanical alloying and powder compaction should be possible.

precipitation, but also preceding quenching below the martensite start temperature. This allowed us to tailor the process parameters to either avoid or provoke in situ precipitation, illustrating how AM processes can be used to digitally control the material microstructure and hence the properties.

To produce ODS steels via AM, our second hypothesis, we blended micron-sized ferritic steel powders with nano-sized yttria powders. Despite process optimisation, production by LMD did not yield the desired results, with all oxides being lost in slag formation. However, after changing our focus to the Selective Laser Melting (SLM) process, which involves solidification rates that are orders of magnitude higher than in LMD, we could retain up to 35% of the introduced yttria, despite some coarsening of oxides. In a separate process route, we introduced the oxide particles

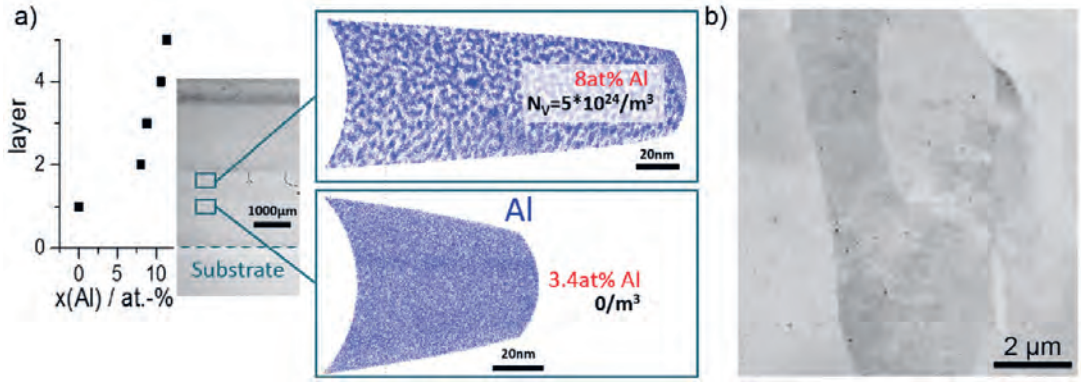


Fig. 1: *a) NiAl precipitates in a Fe-19Ni-xAl compositionally graded model maraging steel produced in situ during Laser Metal Deposition. b) Oxide particles in a ferritic stainless steel (Fe-20Cr-5Al-0.5Ti) produced during Selective Laser Melting in a 8% CO_2 process atmosphere.*

To test the first hypothesis, we produced samples from highly supersaturated maraging steel model alloys. The AM process in use was Laser Metal Deposition (LMD), in which metal powder is blown by a carrier gas into a melt pool generated by a laser beam. By using two powder feeders simultaneously, we varied the composition of the deposited material, producing compositionally graded Fe-19Ni-xAl specimens. Beyond a certain Al concentration, a very high number density of NiAl precipitates occurred in the material (up to $10^{25}m^{-3}$), as evidenced by Atom Probe Tomography and High Energy X-Ray Diffraction. Crucially, this did not require ageing post-heat treatment, but was a direct result of the intrinsic heat treatment, thus proving our hypothesis. Our model alloys showed promising mechanical properties with tensile strengths up to 1.6 GPa. Extending our research to the Fe-Ni-Ti system, we showed that not only re-heating is required to trigger not by a separate powder, but by an *in situ* reaction of the steel matrix with an oxidising gas atmosphere. We successfully produced ODS steel samples both in LMD with a process atmosphere of Ar+3%O_2, as well as in SLM with Ar atmospheres containing from 2% to 100% CO_2.

References

1. Kürnsteiner, P.; Wilms, M.B.; Weisheit, A.; Barriobero-Vila, P.; Jägle, E.A.; Raabe, D.: Acta Mater 129 (2017) 52.
2. Jägle, E.A.; Sheng, Z.; Kürsteiner, P.; Ocylok, S.; Weisheit, A.; Raabe, D.: Materials 10 (2017) 8.
3. Knoll, H.; Ocylok, S.; Weisheit, A.; Springer, H.; Jägle, E.A.; Raabe, D.: Steel Res Int 88 (2017) 1600416.
4. Springer, H.; Baron, C.; Szczepaniak, A.; Jägle, E.A.; Wilms, M.B.; Weisheit, A.; Raabe, D.: Mater Des 111 (2016) 60.
5. Jägle, E.A.; Sheng, Z.; Lu, L.; Wu, L.; Risse, J.; Weisheit, A.; Raabe, D.: JOM 68 (2016) 943.

Initial Wear: Engineering down to the Atomic Scale

Z. Peng [1], B. Gault [1], T. Meiners [2], P.-P. Choi [3], C. Liebscher [2], M. Rohwerder [4], D. Raabe [1], M. Friedrichs [5], O. Dambon [5], F. Klocke [5]

[1] Department of Microstructure Physics and Alloy Design (MA)
[2] Department of Structure and Nano-/ Micromechanics of Materials (SN)
[3] now at: Department of Materials Science & Engineering, Korean Advanced Institute of Science and Technology
[4] Department of Interface Chemistry and Surface Engineering (GO)
[5] Fraunhofer Institute for Production Technology, IPT

Driven by the wide range of applications in laser technology, biomedicine and consumer electronics, etc., the demand for high-quality lenses with complex geometries and small dimensions is rapidly growing. Since direct grinding and polishing of such lenses is not economically competitive, precision glass moulding (PGM) has become a popular manufacturing route. PGM is a replicative technology for efficiently producing high-precision optical lenses in medium or high volumes. PGM is carried out at temperatures ranging from 350 to 800 °C and at a high press force (2–20 kN). The degradation of the moulds is very fast. Production costs are directly related to the moulds' lifetime.

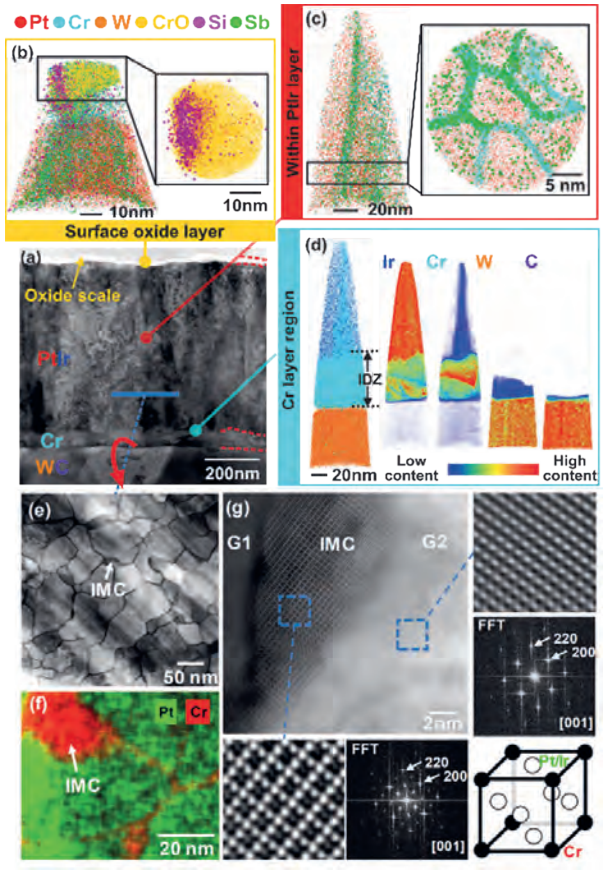


Fig. 1: *Representative characterization results of a mould sample after 120 PGM production cycles: (a) cross-sectional STEM image, (b-d) APT results from different regions of the sample, (e) plane view STEM image of the PtIr layer, (f) STEM-EDX map, (g) HR-STEM image resolving the atomic structure of the intermetallic phase (IMC) and a PtIr grain. As the detailed STEM analysis reveals (e-g)), the diffusion and segregation of Cr along the PtIr grain boundaries also lead to heterogeneous precipitation of the $(PtIr)_3Cr$ intermetallic phase.*

Initial Wear's aim was to gain a fundamental understanding of the degradation mechanisms of the mould materials, so as to inform new design strategies for high-performance and high-durability moulds, which is crucial to the PGM industry. To gain insights into the degradation mechanisms, we applied atom probe tomography (APT) and scanning transmission electron microscopy (STEM). APT has the capability to reveal three-dimensional (3D) chemical information with sub-nanometre precision and a sensitivity in the range of tens of part-per-million. STEM allows for precise structural characterisation.

The samples are made of cemented tungsten carbide covered by 20-nm-thick Cr interlayer to enhance adhesion of the main protective coating made of a 600-nm-thick PtIr layer. In controlled laboratory experiments, we found that the degradation is initiated by Cr segregation at the grain boundaries (GBs) in the PtIr and subsequent diffusion to the surface and formation of an oxide layer [1]. Similar degradation processes were also observed in the technical simulation (see fig.1). We found this can be prevented by decreasing the oxygen partial pressure in the environment. Research is ongoing to investigate the underpinnings of these new observations.

In summary, the key steps of the degradation mechanisms are:

1. outward diffusion of Cr atoms along the grain boundaries of the PtIr layer;
2. oxidation of Cr on the surface;
3. bulk interdiffusion between PtIr, Cr, and the WC substrate;
4. penetration of glass fragments into the mould.

To impede the degradation process, we have also tested Ni as an interlayer material, but it behaved even worse. (see [1] for more details). So decreasing the oxygen partial pressure of the processing chamber should be achieved.

References

1. Peng, Z.; Rohwerder, M.; Choi, P.P.; Gault, B.; Meiners, T.; Friedrichs, M.; Kreilkamp, H.; Klocke, F.; Raabe, D.: Corros Sci 120 (2017) 1.

CarMON - Carbon Metal Oxide Nanohybrids: A Leibniz – Max-Planck Cooperation

S. Hieke [1], A. Frank [1], V. Presser [2], S. Fleischmann [2], A. Kruth [3], M. Dias [3], S. Peglow [3], C. Scheu [1]

[1] Independent Max Planck Research Group on Nanoanalytics and Interfaces (NG)
[2] INM – Leibniz Institute for New Materials, Saarbrücken, and Department of Materials Science, Saarland University
[3] INP – Leibniz Institute for Plasma Science and Technology, Greifswald

The CarMON project – short for Carbon-metal oxide nanohybrids – is a collaborative project funded by the Leibniz Association. The project aims to synthesize novel materials and material combinations on the nanoscale for electrochemical applications. CarMON is connecting the Leibniz Institute for Plasma Science and Technology (INP) in Greifswald, the INM - Leibniz Institute for New Materials in Saarbrücken and the independent Max Planck research group on Nanoanalytics and Interfaces (NG) at the MPIE in Düsseldorf. This way, the expertise of the INP regarding plasma-related synthesis and analysis, the competence of the INM in developing new nanomaterials and the abilities at the MPIE concerning advanced (scanning) transmission electron microscopic ((S)TEM) analysis are linked closely, thus enabling new synthesis concepts for novel nanomaterials.

As humanity is facing global warming and fossil fuels are getting scarce, the need for alternative energy concepts – generation and storage – is ever-present. The collaborators aim to hybridize carbonaceous materials and metal oxides, as a synergistic combination of these materials on the nanoscale leads to enhanced properties for various electrochemical applications. In the current project, the search for nanohybrids for electrode appliance in Li-ion batteries and for desalination of water is in the foreground. However, for blending these materials, traditional chemical methods reach their limits regarding defects and imperfections in the crystals which can decrease their performance. Therefore, new synthesis routes are needed and can be found among plasma-assisted processes offering scalable processes at relatively low temperatures and minimal waste.

It is important to control the synthesis and closely link the procedure to properties and crystallinity of the material. Systematic studies varying the deposition parameters for vanadium oxides on silicon have been performed at the INP. The resulting films have been characterized at the MPIE to achieve a deep understanding of the growth behaviour. High-resolution TEM measurements on a C_s image-corrected Titan Themis allow to judge the crystallinity of the as-prepared films. These findings can be complemented by (nanobeam) electron diffraction studies.

Furthermore, electron energy loss spectroscopy investigations performed in scanning TEM enable the allocation of light elements for MoO_x/MoS_2 core-shell particles on carbon nanotubes prepared at the INM (Fig. 1). The use of the C_s probe-corrected Titan Themis at the MPIE provides site-specific analytics on the nanoscale, proving the core-shell nature of the samples, as electron energy loss spectroscopy allows to also distinguish light elements like oxygen and sulfur.

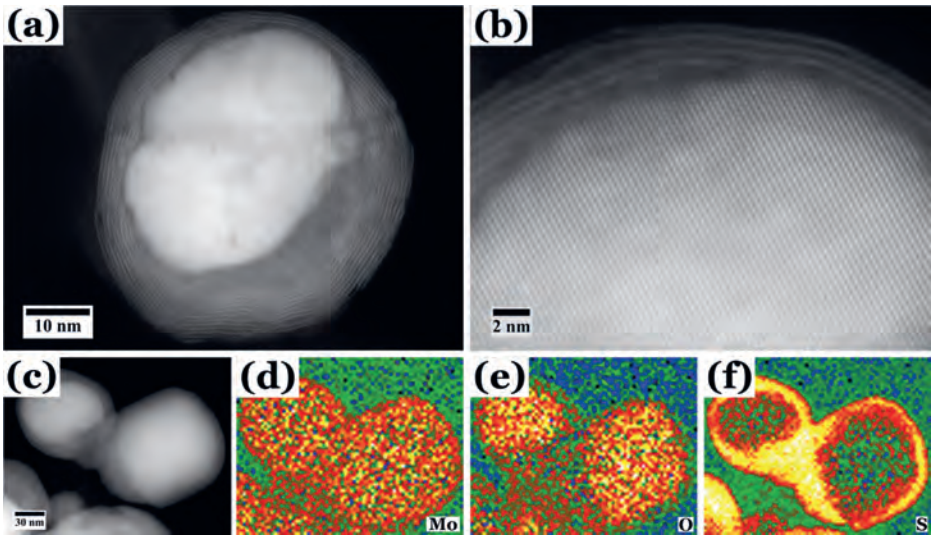


Fig. 1: *(a) HAADF STEM image of one MoO_x / MoS_2 core-shell particle and (b) detail revealing the shell structure, (c) HAADF STEM image of MoO_x / MoS_2 core-shell particles, and (d), (e), (f) corresponding electron energy loss elemental maps of molybdenum, oxygen, and sulfur, respectively.*

International Scientific Coordination Network Mechanics of Nano-Objects - GDRi MECANO

G. Dehm [1], D. Raabe [2], C. Kirchlechner [1]

[1] Department of Structure and Nano-/ Micromechanics of Materials (SN)
[2] Department of Microstructure Physics and Alloy Design (MA)

The International Scientific Coordination Network (GDRi) Mechanics of Nano-Objects (MECANO) is a multinational network initiated by Centre National de la Recherche Scientifique (CNRS) in France. This essentially European network includes 31 French partners (including 4 industrial ones) and 11 laboratories from Germany, Austria, Italy, Switzerland, UK and Ireland. The common ground on which all the partners are working on are condensed matter objects that possess at least one internal or external dimension smaller than a micron. Size effects can appear on various properties due to this structural confinement. However, mechanical size effects are at the core of the MECANO network. GDRi MECANO, which was established in 2012 by Prof. Olivier Thomas (U Marseilles) and is now in its second phase (2016-2019) led by Dr. Marc Legros (CNRS-CEMES Toulouse) brings together physicist, chemists, materials scientists and mechanical engineers. G. Dehm is a member of the Scientific Committee, which meets annually to decide on topics of forthcoming activities. The formation of early career scientists is certainly one of the most important goals that this network aims at.

The scientific questions addressed within MECANO stretch from the 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 (continuums mechanics, plasticity, elasticity theory, …) can be applied to smaller and smaller length scales.

The GDRi 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. C. Kirchlechner from the MPIE organized a summer school on “Experimental Nano- and Micromechanics” in 2017 with B. Merle (U Erlangen). The school included hands-on exercises in the laboratories at the MPIE comprising nanoindentation, electron backscatter diffraction, electron channelling contrast imaging, microfracture mechanics, and thin film mechanics. A general GDRi MECANO school was held at 28 Oct. - 2. Nov. 2018 in France with G. Dehm teaching a course on stresses and plasticity in thin films. The GDRi MECANO network is very fruitful in fostering collaborations and exchanges between its partners and the young researchers.

Fig. 1: *Participants of the summer school “Experimental Nano- and Micromechanics” held at the MPIE in Düsseldorf from 11-15 September 2017.*

Development of Cost-Efficient, High-Performance Gas Diffusion Electrodes for Polymer Electrolyte Membrane Fuel Cells with Low Platinum Loading and Newly Developed Hollow Graphitic Spheres as Support Material

M. Ledendecker [1], D. Göhl [1], K.J.J. Mayrhofer [1,2,3]

[1] Department of Interface Chemistry and Surface Engineering (GO)
[2] Helmholtz Institute Erlangen-Nürnberg for Renewable Energy
[3] Friedrich-Alexander-Universität Erlangen-Nürnberg

The main goal in catalysis is the development of active, stable and selective catalysts while keeping the material and production costs low. In recent years, the understanding of fundamental catalytic processes has led to considerable improvement in catalyst design with more active and stable catalysts and supports. The joint project comprises different partners in order to develop catalyst/support design strategies from a fundamental, experimental and application point of view and is aimed to establish active and most importantly highly stable fuel cell catalysts and electrodes. The catalyst synthesis and optimization is performed in close collaboration with Prof. Ferdi Schüth at the Max-Planck-Institut für Kohlenforschung (MPI-KOFO). Through the concept of pore-confinement, more stable catalysts towards the oxygen reduction reaction are developed. Together with the Max-Planck-Institut für Chemische Energiekonversion (MPI-CEC) and Ernst Ruska-Centre (ERC)-Jülich, the MPIE as the project leader focuses on mechanistic studies for dissolution and structural degradation of supported nanoparticles. We establish potential boundary conditions for the desired application, point out degradation pathways and develop strategies to overcome stability challenges [1-4]. Fully automated rotating disk electrode (RDE) measurements and the scanning flow cell (SFC) coupled to an inductivly coupled plasma - mass spectrometer (ICP-MS) are the working horses for the in depth characterization (Fig. 1), which are further complemented by HR-STEM, XRD, XPS and atomistic modelling. Promising catalysts are investigated further at the Zentrum für BrennstoffzellenTechnik GmbH in Duisburg and with material technology partners from industry. Moreover, scale-up strategies for synthesis are developed for high performance catalysts in order to pave the path for large-scale applications.

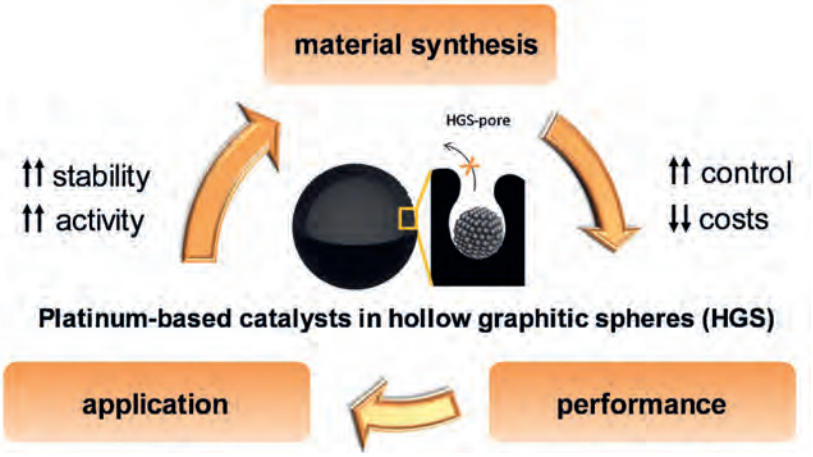


Fig. 1: *Overview of the "PtTM@HGS" project. The focus at the MPIE lies in benchmarking catalysts regarding activity, stability and on the elaboration of fundamental processes taking place during catalysis.*

The research project is funded by the Federal Ministry for Economic Affairs and Energy (BMWi) of Germany in the framework of PtTM@HGS (project number 03ET6080A).

References

1. Pizzutilo, E.; Knossalla, J.; Geiger, S.; Grote, J.P.; Polymeros, G.; Baldizzone, C.; Mezzavilla, S.; Ledendecker, M.; Mingers, A.; Cherevko, S.; Schüth, F.; Mayrhofer K.J.J.: Adv Energy Mater. 7 (2017) 7 1700835.
2. Göhl, D.; Mingers, A.M.; Geiger, S.; Schalenbach, M.; Cherevko, S.; Knossalla, J.; Jalalpoor, D.; Schüth, F.; Mayrhofer, K.J.J.; Ledendecker, M.: Electrochim Acta. 270 (2018) 70.
3. Baldizzone, C.; Swertz, A.-C.; Hodnik, N.; Pizzutilo, E.; Polymeros, G.; Keeley, G. P.; Knossalla, J.; Heggen, M.; Mayrhofer, K.J.J.; Schüth, F.: ACS Catal. 6 (2016) 8058.
4. Knossalla, J.; Paciok, P.; Göhl, D.; Jalalpoor, D.; Pizzutilo, E.; Mingers, A.M.; Heggen, M.; Dunin-Borkowski, R.; Mayrhofer, K.J.J.; Schüth, F.; Ledendecker, M.: J Am Chem Soc. 140 (2018) 15684.

Compositionally Complex Alloys – High Entropy Alloys (CCA-HEA)

F. Körmann [1], Y. Ikeda [1], J. Neugebauer [1], Z. Li [2], D. Raabe [2], E. A. Jägle [2], G. Dehm [3], C. Liebscher [3]

[1] Department of Computational Materials Design (CM)
[2] Department of Microstructure Physics and Alloy Design (MA)
[3] Department of Structure and Nano-/ Micromechanics of Materials (SN)

The recently introduced novel class of materials, referred to as "high entropy alloys" (HEA) for single phase materials or "compositionally complex alloys" (CCA) in case of 2 or more phases contain multiple principal elements and fundamentally differ from conventional alloys with one or two prevalent base elements. This new CCA-HEA concept has opened up a huge compositional space of multicomponent alloys with significant technological potential but also posing new and exciting scientific challenges. The German Research Foundation (DFG) has established in 2017 the Priority Programme "Compositionally Complex Alloys – High Entropy Alloys" to develop CCAs and HEAs with outstanding mechanical properties or materials behaviour.

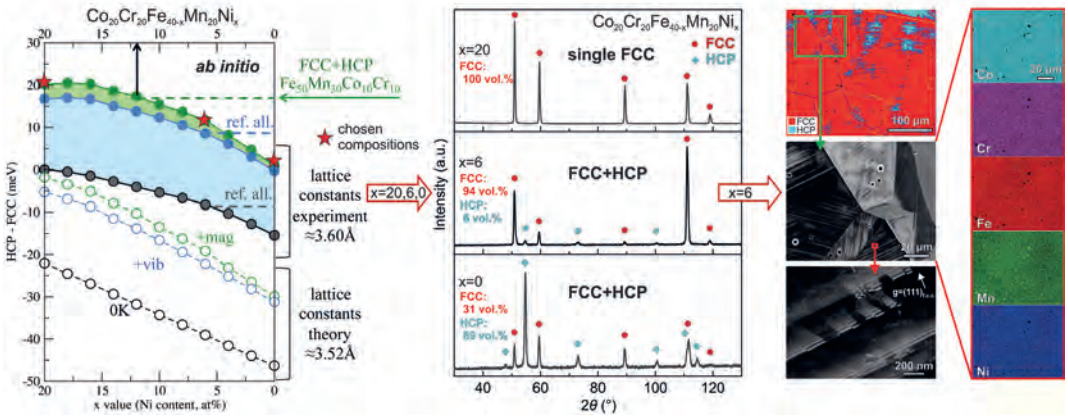


Fig. 1: *From left to right: ab initio study of FCC-HCP stability of $Co_{20}Cr_{20}Fe_{40-x}Mn_{20}Ni_x$ alloys, XRD patterns for three HEAs/CCAs with x=20, 6 and 0 at.%, microstructure and elemental distribution of $Co_{20}Cr_{20}Fe_{34}Mn_{20}Ni_6$ [1].*

body-centred cubic CCAs with tailored precipitates based on B2- and/or $L2_1$-phases. In a first step, the phase formation in the rather unexplored composition space of FeAlCr (Mn, Co, Ni, Ti) is explored by high throughput screening and characterization based on thin film deposition techniques. Promising alloy candidates are identified fulfilling the conditions to have a BCC crystal structure and showing either B2, $L2_1$ or both types of precipitate phases followed by a microstructural optimization in terms of precipitate morphology, coherency with the supersaturated BCC matrix and their volume fraction.

(3) "Particle-strengthened Compositionally Complex Alloys - interlinking powder synthesis, additive manufacturing, microstructure evolution and deformation mechanisms" (E. Jägle, G. Dehm, V. Uhlenwinkel/Bremen). Even though it is known that many CCAs contain nano-scaled particles, it is not yet understood if their interaction with gliding dislocations gives rise to strengthening effects following the same laws as for conventional alloys. In this project, the material synthesis follows a powder metallurgical route via gas atomization and additive manufacturing of specimens, allowing rapid compositional screening and minimum inhomogeneity. The research questions will be tackled by investigating nitride and B2-strenghtened FeNiCoCr(Al,Mn)-alloys by a combination of high-resolution analysis employing transmission electron microscopy and atom probe microscopy, mechanical testing and quantitative *in situ* micro-mechanical experiments.

The MPIE is strongly represented in this SPP with three granted projects:

(1) "Interstitial transformation-induced plasticity-assisted quinary compositionally complex alloys: Design, structure and mechanical behaviour (TRIP-iCCAs)" (F. Körmann, Z. Li, D. Raabe). This project proposes a new class of CCAs, namely, interstitially alloyed TRIP-assisted quinary CCAs. Due to the TRIP effect the alloys will have dual-phase structure [1] after strain loading. Interstitial-free quinary CCAs with TRIP effect [2] will first be developed followed by alloying with carbon and nitrogen. This enables the further tuning of the alloy's strain-hardening ability and the introduction of interstitial solid solution strengthening effect. The project employs state-of-the-art theoretical *ab initio* simulation and experimental techniques.

(2) "Tailored precipitation strengthened, compositionally complex FeAlCr (Mn, Co, Ni, Ti) alloys for high temperature applications" (C. Liebscher, K. Pradeep / IIT Madras). This project aims to develop

References

1. Li, Z.; Pradeep, K.G.; Deng, Y.; Raabe, D.; Tasan, C.C.: Nature 534 (2016) 227.
2. Li, Z.; Körmann, F.; Grabowski, B.; Neugebauer, J.; Raabe, D.: Acta Mater 136 (2017) 262.

Advanced Combinatorial Alloy Design: High Throughput for Evolutionary Structural Materials

H. Springer, D. Raabe

Department of Microstructure Physics and Alloy Design (MA)

The collaborative research centre (SFB) 1232 “Farbige Zustände (coloured states)” targets the establishment of a high-throughput methodology for the accelerated development of innovative structural materials with novel property profiles, as well as their prediction using a heuristic model (Fig. 1). This knowledge based design is relying on systematic investigations neccessary to describe the underlying relationships in the form of so called descriptors. For that purpose, small, fast and effective methods for synthesis and thermo-mechanical processing are being developed and deployed. Based on simplified tests of the mechanical and chemical behaviour, evolution-based search methods are utilised to derive so called predictor functions to forecast the more complex material properties relevant for engineering, such as the stress/strain behaviour during tensile testing.

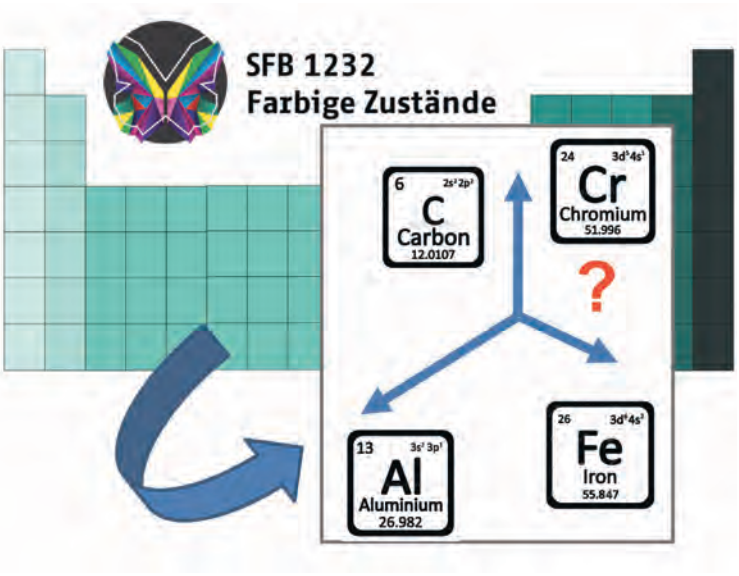


Fig. 1: *CRC1232: Identifying and developing innovative structural materials by exploring new ranges of chemical compostions and thermo-mechanical processing parameters.*

A critical point in this high throughput methodology based on small material volumes lies in the necessary transferability of the derived results to industrial volumes of production. This is the one of two objectives for the sub-project “Validation” from the MPIE within the framework of the CRC. The influence of rapid solidification – which the small material volumes in the order of milligrams are subjected to – on microstructures and mechanical properties is investigated with sprayforming techniques, together with partners at the Leibniz-Institut für Werkstofftechnik in Bremen. Furthermore, bulk high throughput methods developed and established at the MPIE, termed rapid alloy prototyping (RAP), are utilised [1]. The highly parallised and standardised sequence of RAP regarding synthesis, processing and characterisation is used to systematically screen the wide range of materials states achievable through chemical and thermo-mechanical variations. Together with literature data, these investigations provide a solid basis for the methodology development. The chosen alloy system Fe–xC–yCr (x = 0 – 1, y = 0 – 20) is especially suited for this purpose due to the numerous complex microstructural phenomena taking place on various scales: Depending on the respective thermo-mechanical variation, mechanisms such as martensitic transformation, austenite reversion, carbon partitioning or carbide precipitiation to name but a few, can be exploited for a vast range of mechanical and chemical properties [2,3]. Additionally to valididating the high throughput methodology, the vast range of investigated materials also offers the unique opportunity to identify yet unknown property combinations. Elucidating their microstructural origins with high resolution characterisation techniques represents the second objective of our sub-project. The MPIE is further involved in the CRC by heading the work group “Suchraumdefinition”, which is concerned with providing metallurgical knowledge to projects more concerned with process engineering, and helps defining the strategic direction in which the material development is headed.

References

1. Springer, H.; Raabe, D.: Acta Mater 60 (2012) 4950.
2. Springer, H.; Belde, M.M.; Raabe, D.: Mater Sci Eng A 582 (2013) 235.
3. Springer, H.; Belde, M.M.; Raabe, D.: Mater Design 90 (2016) 1100.

Collaborative Research Centre TRR 188: Damage is not Failure

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

[1] Department of Structure and Nano-/ Micromechanics of Materials (SN)
[2] Department of Microstructure Physics and Alloy Design (MA)

The formation of voids and micro-cracks during plastic deformation of ductile materials occurs not only during service, but also already during the manufacture of engineering components. Typically, the damage imposed during manufacture is quantitatively unknown, which results in uncertainties in prediction of lifetime and defining safety factors. Thus, within the Collaborative Research Centre TRR 188 we want to understand, predict and control the damage evolution during forming of engineering components. The ultimate goal of the research centre is to cut the costs during development of forming parts, to increase the safety and reliability and, concomitantly, reduce the weight of components significantly. The interdisciplinary research strategy comprises mechanical engineers, experts in forming technology, in solid state mechanics and in microstructural physics located at the RWTH Aachen University, at the TU Dortmund and the MPIE. The approach bridges from the micrometre regime (microstructural length scale) up to metres (sample length scale).

At the MPIE we are measuring the local mechanical properties of a dual phase steel (DP800). Besides the size dependent single-phase properties of ferrite, martensite and non-metallic inclusions, we are also developing mechanism-based material laws for interface fracture of the ferrite-martensite interface. The first surprising result shows that the critical resolved shear stress (CRSS) of ferrite is similar for slip on the (110), (112) and (123) slip plane (Fig. 1). These results will be implemented in the crystal plasticity finite element simulations of the cooperating research groups within the TRR188.

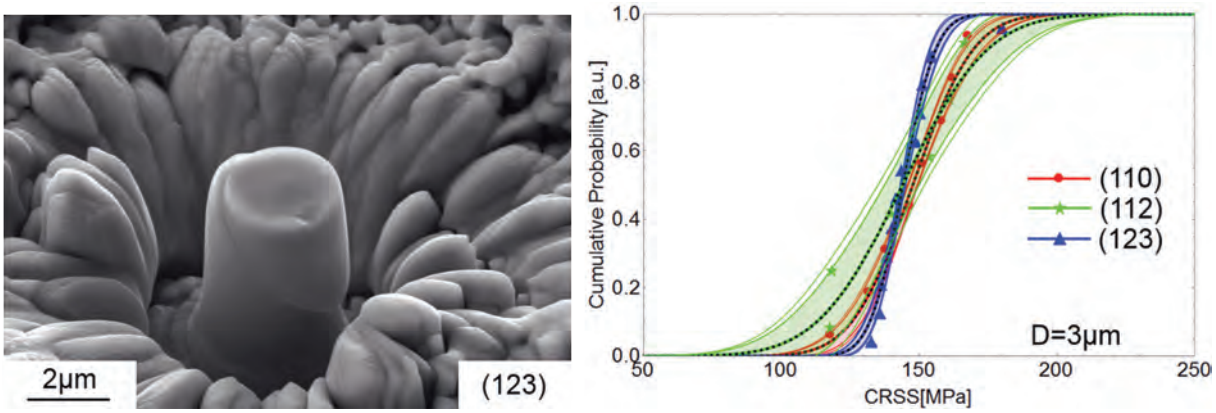


Fig. 1: *Ferrite micropillar exhibiting slip on the (123) slip plane. The cumulative probability of the critical resolved shear stress (CRSS) is shown for all three families of slip plane.*

Gas Turbines of Tomorrow: From Atom to Turbine Blade

S.K. Makineni [1], J. He [1], P. Kontis [1], X. Wu [2, 3], M. Lenz [4], E. Spiecker [4], S. Neumeier [4], G. Dehm [5], D. Raabe [1], R. Reed [6, 7], J. Cormier [8], G. Eggeler [2, 3], B. Gault [1]

[1] Department of Microstructure Physics and Alloy Design (MA)
[2] Material Science and Engineering, Ruhr-Universität Bochum
[3] Max Planck Group on High Temperature Materials
[4] Institute of General Materials Properties, Friedrich-Alexander Universität Erlangen-Nürnberg
[5] Department of Structure and Nano-/ Micromechanics of Materials (SN)
[6] Depts. of Engineering Science and Materials, University of Oxford
[7] Alexander von Humboldt Awardee at the MPIE
[8] Institut PPRIMME, CNRS, ENSMA & Université de Poitiers, France

The atom probe tomography (APT) group in the MA department is very active in high-temperature materials, with a strong emphasis on Ni- and Co-based superalloys. Within the second phase of the DFG-funded SFB/TR 103 "From atoms to turbine blades – a scientific basis for a new generation of single crystal superalloys" joint research centre with Ruhr-Universität Bochum and Friedrich-Alexander-Universität Erlangen-Nürnberg, we established a method to facilitate correlative electron microscopy and atom probe tomography [1]. Here, specimen preparation is guided by controlled-electron channelling contrast imaging [2], as seen in Fig. 1. Deployed to investigate the mechanisms of creep deformation in new CoNi-based superalloys, this approach provided insights into the diffusional processes that limit the rate of deformation [3,4]. In Ni-based alloy ERBO-1, we demonstrated that Re was carried by moving dislocations during creep and loses its strengthening effect, thereby likely providing answers to a long-standing debate regarding the role of Re in enhancing the alloy's creep performance.

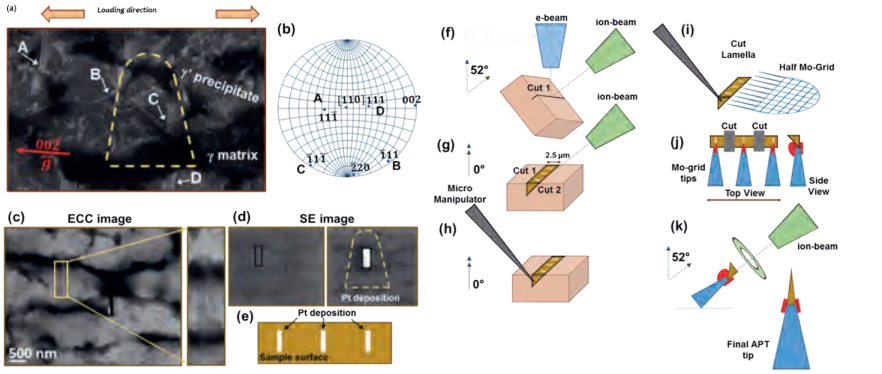


Fig. 1: *Complete methodology for targeted specimen preparation from specific crystalline defects using controlled-electron-channelling contrast imaging, transmission electron microscopy and atom probe tomography.*

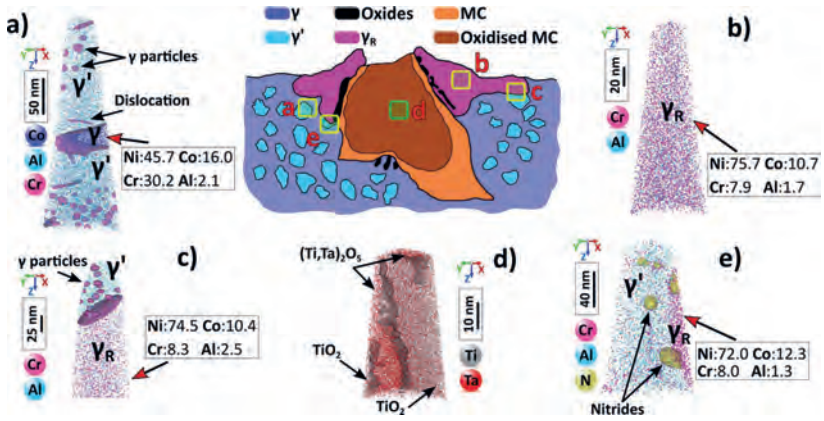


Fig. 2: *Summary of APT microstructural characterisation from the surroundings of an oxidised carbide in a multicrystalline Ni-based superalloy [6].*

In parallel, we established new international collaborations with the University of Oxford (UK) [5,6] and the Institut P' a CNRS-laboratory in Poitiers [5] on commercial single crystal and multi-crystalline Ni-based superalloys. We provided evidence of mass transport of specific solutes by or through defects generated during creep or because of the oxidation of intergranular carbides [7]. The possibility that mechanisms such as pipe diffusion could affect high temperature mechanical deformation had been proposed over a decade ago, but we provided the first experimental proof that this was the case, and provided an explanation for how this could affect the local equilibrium and hence the strengthening of the alloy.

Overall, our investigations have thrown light onto the interplay between structural defects and local composition, and hence stability of the alloys in service. Understanding these fundamental aspects is critical to explore new possible alloy design routes to enhance the lifetime of parts, increase operating temperatures and so as to reduce fuel consumption, with clear environmental gains.

References

1. Makineni, S.K.; Lenz, M.; Kontis, P.; Li, Z.; Kumar, A.; Felfer, P.J.; Neumeier, S.; Herbig, M.; Spiecker, E.; Raabe, D.; Gault, B.: JOM 70 (2018) 1736.
2. Zaefferer, S.; Elhami, N.N.: Acta Mater 75 (2014) 20.
3. Makineni, S.K.; Kumar, A.; Lenz, M.; Kontis, P.; Meiners, T.; Zenk, C.; Zaefferer, S.; Eggeler, G.; Neumeier, S.; Spiecker, E.; Raabe, D.; Gault, B.: Acta Mater 155 (2018) 362.
4. Makineni, S.K.; Lenz, M.; Neumeier, S.; Spiecker, E.; Raabe, D.: Scr Mater 157 (2018) 62.
5. Kontis, P.; Li, Z.; Collins, D.M.; Cormier, J.; Raabe, D.; Gault, B.: Scr Mater 145 (2018) 76.
6. Kontis, P.; Collins, D.M.; Wilkinson, A.J.; Reed, R.C.; Raabe, D.; Gault, B.: Scr Mater 147 (2018) 59.
7. Kontis, P.; Li, Z.; Segersäll, M.; Moverare, J.J.; Reed, R.C.; Raabe, D.; Gault, B.: Metall Mater Trans A 49 (2018) 4236.

Metallic Nanowires on the Atomic Scale: Electronic and Vibrational Coupling in Real World Systems

S. Wippermann

Department of Interface Chemistry and Surface Engineering (GO)

In 2015, S. Wippermann, head of the Atomistic Modeling Group, joined the FOR 1700 research unit funded by the German Research Foundation (DFG) [1] as a principal investigator. Ideal one-dimensional (1D) electronic systems show fascinating physical properties, such as quantization of conductance, charge density waves (CDW), non-Fermi liquid behaviour and often a variety of instabilities with associated phase transitions. The central topic of the FOR 1700 research group is the exploration and identification of physical systems with 1D electronic properties under explicit consideration of 2D and 3D coupling, their control and manipulation.

These findings are of particular interest because the (8x2) CDW state features associated non-trivial topological electronic properties. The topological properties of this system give rise to fundamental excitations of the CDW, which take the form of topological solitons. Three different types of solitons are observed, carrying a charge of exactly either +1, 0 or -1 electrons, respectively. Soliton-soliton interactions were shown to be topologically protected and to obey an Abelian group. In principle, these fundamental excitations of the CDW can be used as information carriers in new types of information processing. Exactly those electronic states whose excitation

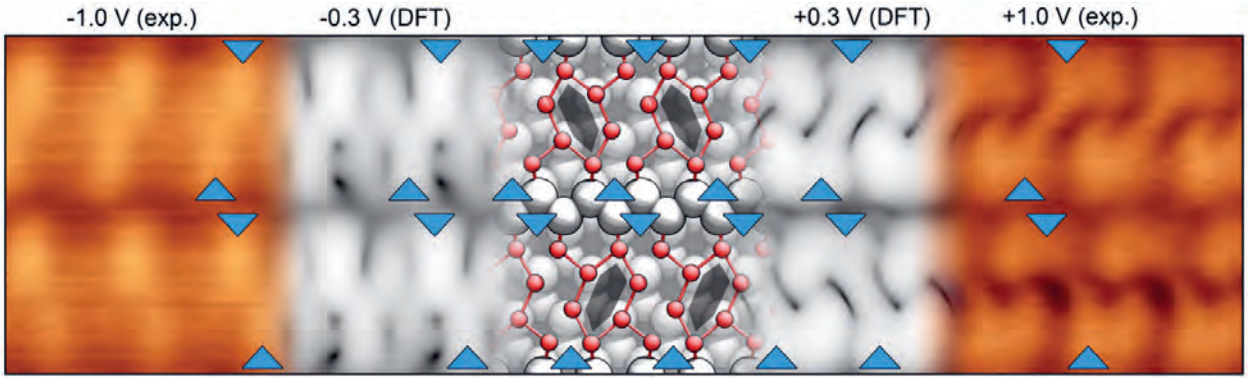


Fig. 1: *Structural model of the (8x2) CDW ground state, overlayed with scanning tunnelling microscopy (STM) measurements and density functional theory (DFT) simulations (STM measurements: T. H. Kim, H. W. Yeom, Pohang University of Science and Technology). Red balls and grey spheres represent indium and silicon atoms, respectively.*

The ordered array of indium atomic wires formed at the Si(111)-(4x1)In surface is a particularly interesting model system of this kind. It features a reversible temperature-induced metal-insulator transition at $T < T_C = 120$ K into a CDW ground state with (8x2) translational symmetry. Due to strongly coupled electronic and lattice degrees of freedom, femtosecond laser excitation can induce a non-thermal transition from the semiconducting (8x2) CDW ground state into a metallic supercooled (4x1) state. This transition is completed within 350 fs. We demonstrated that carefully tuned electronic excitations can create non-equilibrium potential energy surfaces that drive structural dynamics at the quantum limit, that is, in a regime where the nuclear motion is directed and deterministic [2].

can drive the transition from the (8x2) into the (4x1) state [2] also couple strongly and selectively to the topological solitons. Currently, we are exploring in detail electron-phonon, electron-soliton and soliton-soliton interactions.

References

1. http://www.atomicwires.de
2. Frigge, T.; Hafke, B.; Witte, T.; Krenzer, B.; Samad Syed, A.; Miksic Trontl, V.; Avigo, I.; Zhou, P.; Ligges, M.; von der Linde, D.; Bovensiepen, U.; Horn von Hoegen, M.; Wippermann, S.; Lücke, A.; Sanna, S.; Gerstmann, U.; Schmidt, W.G.: Nature 544 (2017) 207.

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

T. Hickel [1], D. Ponge [2], M. Herbig [2], B. Gault [2], S. Zaefferer [2], F. Roters [2], C. Scheu [3], J. Neugebauer [1], D. Raabe [2]

[1] Department of Computational Materials Design (CM)
[2] Department of Microstructure Physics and Alloy Design (MA)
[3] Independent Max Planck Research Group on Nanoanalytics and Interfaces (NG)

In 2007 the Max Planck Institute (MPIE) 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 (DFG). Speaker and chairman of the project is Prof. Wolfgang Bleck (Institute of Ferrous Metallurgy, RWTH) and vice-chairman is Prof. Dierk Raabe (MPIE). Due to several successful evaluations, the project is now running in the third four-year funding period, which started in July 2015. In this period, the MPIE is heading seven partial projects, involving scientists from three departments.

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 atomic structure simulations and engineering applications. 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 MPIE contributes with theory and experiments to the success of this project. *Ab initio* methods are not only used for the prediction of key thermodynamic parameters, but turned out to become increasingly important for understanding the different strengthening mechanisms (TWIP, TRIP, shear band formation). In combination with constitutive models, the extraordinary deformation behaviour with high strain hardening values can even be quantitatively predicted. On the experimental side, in particular correlative studies that combine high-resolution electron microscopy and atom probe tomography provided a combined knowledge of the local structure and chemistry on the atomic scale. This technique was, for example used, to resolve the formation of kappa-carbides, since their superstructure arrangement yields an additional strengthening mechanism in these materials.

The new methods and insights developed within the SFB 761 have an impact beyond the project consortium. A couple of transfer projects are established to allow industrial partners to benefit from these results. One of them, which is run by the MPIE, evaluates the sensitivity of high-Mn steels that contain Cr carbides to hydrogen embrittlement. Additionally, the SFB 761 has initiated a series of international conferences on medium- and high-Mn steels. The 4th edition of this conference will take place in April 2019 in Aachen and is co-organized by the MPIE.

More details on SFB761 are available on http://www.stahl-abinitio.de.

Fig. 1: *The SFB 761 team during a meeting in June 2018.*

Activities within the Cluster of Excellence "Ruhr Explores Solvation" (RESOLV)

M. Todorova [1], M. Rohwerder [2], A. Erbe [2], J. Neugebauer [1]

[1] Department of Computational Materials Design (CM)
[2] Department of Interface Chemistry and Surface Engineering (GO)

The Cluster of Excellence RESOLV (Ruhr Explores Solvation Science) was established in November 2012 at the Ruhr-Universität Bochum (RUB). Prof. Dr. Martina Havenith-Newen (Physical Chemistry 2, RUB) is speaker of the cluster in which several departments from RUB, groups from TU Dortmund, University Duisburg-Essen, Fraunhofer UMSICHT Oberhausen, Max-Planck-Institut für Kohlenforschung (Mülheim a. d. Ruhr), Max-Planck-Institute for Chemical Energy Conversion (Mülheim a. d. Ruhr) and the MPIE participate.

With their expertise in interface science, two departments of the MPIE, "Interface Chemistry and Surface Engineering" (GO) and "Computational Materials Design" (CM), are strongly involved in research area C of RESOLV "Ion Solvation and Charge Transfer at Interfaces". Most projects with MPIE involvement call for a strong collaboration between experimentalists and theorists. Two RESOLV-doctoral students, whose main working focus was at the GO department, graduated during the reporting period. One of them worked on a project, which helped to understand the effect of the electric double layer structure on tribology phenomena by force measurements. The second one used interface vibrational spectroscopy to investigate the electrode potential triggered de-solvation of a germanium electrode during a surface transformation. An almost completed third project focuses on the defect evolution in zinc oxide growing on zinc, and the defect formation on ZnO single crystals. Fundamental work in progress investigates the interfacial structure and energy level alignment at immersed electrodes. (see p. 107). The project based in the CM department focuses on the impact of the solvent on the surface structure. Using *ab initio* techniques it could show that solvents have little impact on metallic surfaces but hugely impact semiconducting and insulating surfaces (see p. 89). Ongoing work is addressing questions related to nano-catalysts in wet environments, internal polarisation effects and surface reactivity.

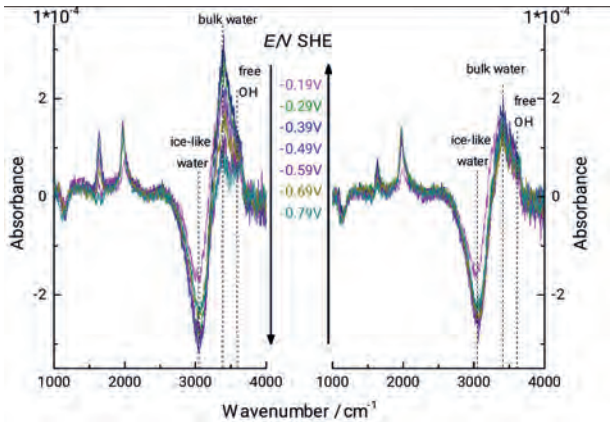


Fig. 1: *Infrared difference absorbance spectra of interfacial water at Ge(100) in contact with 0.1 M $HClO_4$ at different potentials. At the lowest electrode potentials, the surface is H-terminated, which interrupts the hydrogen-bond network to the interface. As a result, the vibrational mode of non-hydrogen bound OH is observed at ~3750 cm^{-1} [1].*

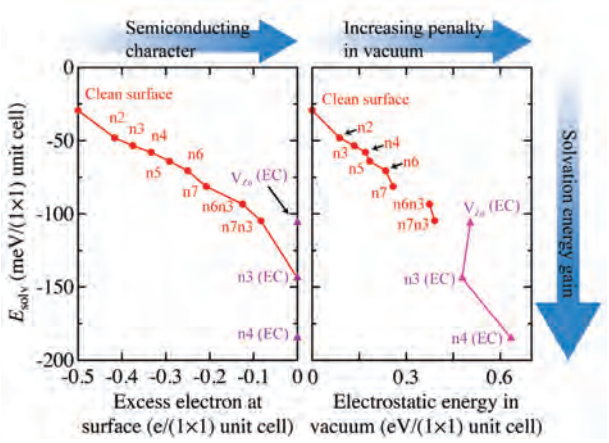


Fig. 2: *Solvation energies of ZnO(0001)-Zn surface structures plotted as a function of (left) the excess electrons at the surface and (right) the electrostatic energy of surface phases referenced to that of the clean surface. In contrast to structures with metallic character, structures with semiconducting character, in particular those with a high electrostatic penalty in vacuum, experience a strong energy gain due to solvation [2].*

In 2017, the Cluster of Excellence RESOLV applied for an extension for a second funding period. The submitted proposal outline was one of the 88 proposals (out of 195 submitted) which were successful. The presentation and defence of the subsequently submitted full proposal took place in Berlin in June 2018, with Prof. Neugebauer (CM) being one of the 15 RESOLV PIs participating in the event. The decision that RESOLV is one of 57 Clusters of Excellence to receive funding for the next 7 years was announced in September 2018.

More details about RESOLV are found at https://solvation.de/

References

1. Niu, F.; Schulz, R.; Medina, A.C.; Schmid, R.; Erbe, A.: Phys Chem Chem Phys 19 (2017) 13585.
2. Yoo, S.H.; Todorova, M.; Neugebauer, J.: Phys Rev Lett 120 (2018) 066101.

Interdisciplinary Centre for Advanced Materials Simulation – ICAMS

J. Neugebauer [1], T. Hickel [1], D. Raabe [2]

[1] Department of Computational Materials Design (CM)
[2] 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 Modeling and Simulation (headed by R. Drautz), Scale Bridging Thermodynamic and Kinetic Simulation (I. Steinbach), and Micromechanical and Macroscopic Modeling (A. Hartmaier) (Fig. 1). After the initial startup period of five years with substantial financial support from an industrial consortium, the state of North Rhine-Westphalia and the European Union, the institute has now become a regular unit within the Ruhr University Bochum and beyond. It serves as an integral part of the Material Research Department within the university, but also of the Materials Chain of the University Alliance Ruhr. The latter combines the research activities in the field of materials science and technology at the three Ruhr Area universities Ruhr-Universität Bochum, TU Dortmund University, and the University of Duisburg-Essen.

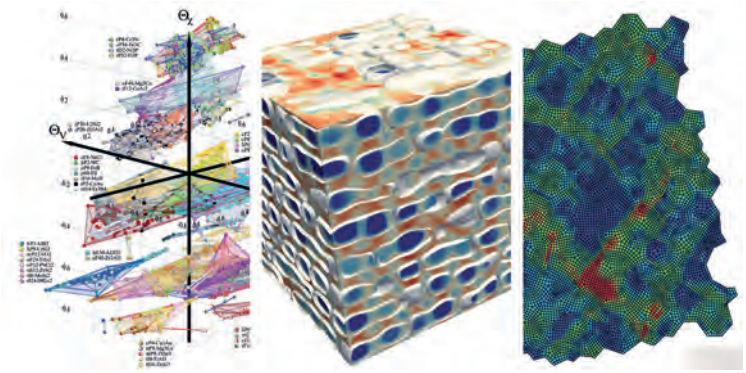


Fig. 1: *The three departments of ICAMS combine simulations at different scales, resulting into (from left) atomic structure maps, phase field and finite element simulations as typical images.*

Structurally, the MPIE is embedded into ICAMS via the Advanced Study Group (ASG) “Modeling”, which supports the multiscale concept in particular at the most fundamental scale, which is dominated by electronic interactions and individual atomic processes. The link between ICAMS and the MPIE is further deepened within two large-scale projects supported 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 Program (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 program “Materials Science and Simulation” (MSS) are partially held by members of the MPIE.

Fig. 2: *Participants at the 10 Years ICAMS International Symposium, taking place 25-27June 2018 in Bochum. The directors Ralf Drautz, Alexander Hartmaier, and Ingo Steinbach (from left) are standing in the first row.*

The strong collaboration between the MPIE and ICAMS is documented by a large number of publications, a significant amount of attracted third-party funding, but in particular by its scientific outreach. Several workshops (e.g. Ringberg Unary Workshop 2013, ADIS workshops 2014, 2016, 2018) and symposia at international conferences are jointly organized with the MPIE. Regular retreats are performed together with the CM department. The ICAMS Advanced Discussions are meanwhile an established institution. The 10 Years ICAMS International Symposium in June 2018 (Fig. 2), belonging to this series of events, gave a perfect chance to look back at a very successful decade and to look forward to many more years of exciting materials research.

Knowledge and Technology Platform for Prediction of Durability and Lifetime of Organic Coated Metals under Long-Term Environmental Corrosion

M. Rohwerder

Department of Interface Chemistry and Surface Engineering (GO)

This is a joint project with the Free University of Brussels (VUB), within the framework of the Research Foundation - Flanders (FWO), in close contact with about twenty cooperating partners from industry, which started operationally in September 2018 at the MPIE.

The motivation behind this project is that at the current state of the art, lifetime and aging assessment of coated metal are performed by means of experimental testing, combining accelerated testing and field testing.

The limitation of the first is that the conditions of the accelerated testing are not representative for the real environmental conditions and that there is no proven relationship between accelerated laboratory testing and field performance data. The limitation of the second is that it takes several years (5 to 10 years). This seriously impedes organic coating development.

The only way to realize a substantial decrease in the development time of new materials is to introduce modelling in the design cycle. What is needed to realize a breakthrough in this field is a tool that can predict quantitatively and dynamically the corrosion behaviour of organic coated metals.

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

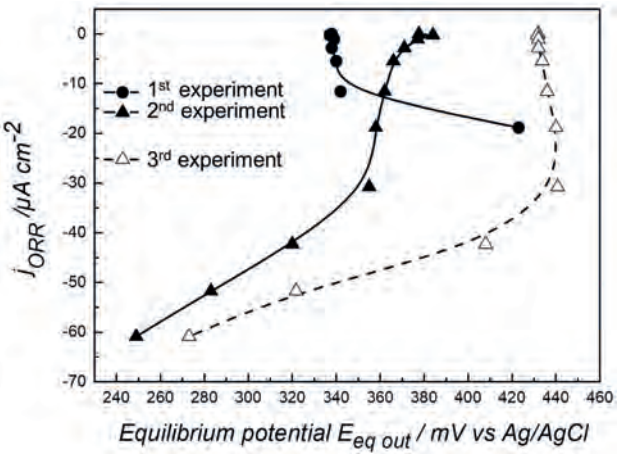


Fig. 1: *Example for I(U) curves obtained at a buried interface: three subsequent I(U) curves obtained on PVB/Pd model samples. In the first scan increasing interfacial degradation results in increasing potentials despite increasing cathodic current density. In the subsequent runs further degradation occurs.*

One task will be a standard characterization of delamination behaviour and the migration of cations at the coating/metal interface. Of crucial importance for this will be a recently developed hydrogen permeation based potentiometric method for obtaining full I(U) curves for oxygen reduction at buried interfaces [1,2]. By observing time dependent changes in the kinetics also information about the corresponding interfacial degradation can be assessed (see fig.1).

References

1. Vijayshankar, D.; Tran, T.H.; Bashir, A.; Evers, S.; Rohwerder, M.: Electrochim Acta 189 (2016) 111.
2. Vijayshankar, D.; Altin, A.; Merola, C.; Bashir, A.; Heinen, E.; Rohwerder, M.: J Electrochem Soc 163 (2016) C778.

Research Awards

ERC Advanced Grant: GB-CORRELATE: Correlating the State and Properties of Grain Boundaries

G. Dehm

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

In August 2018 the new ERC Advanced Grant dedicated to Prof. G. Dehm started with the research project GB-CORRELATE. Grain boundaries (GB) are omnipresent defects in structural and most functional materials. While they are known to strongly influence the electrical, magnetic, thermal or mechanical properties of materials, surprisingly little is known on grain boundary phases, their transitions and how they can be applied to advance materials' design. This is in stark contrast to bulk and surface phases and their transitions - i.e. changes in the arrangements of atoms in the volume or at the surface of materials - where thermodynamic and atomistic concepts are well established since a long time. This lack of a complete exploration of grain boundaries is due to their structural and chemical complexity, their internal defects and previously, insufficient characterization techniques. GB-CORRELATE is tackling these challenges by combining correlated atomic resolved microscopy, spectroscopy and tomography with computational approaches. GB-CORRELATE finds perfect scientific conditions at the MPIE due to the strong in-house collaborations between the different departments.

The complexity of grain boundaries with their large number of macroscopic and microscopic degrees of freedom is approached by a hierarchical strategy: dedicated bicrystals in form of bulk materials and thin films are the reference point to identify

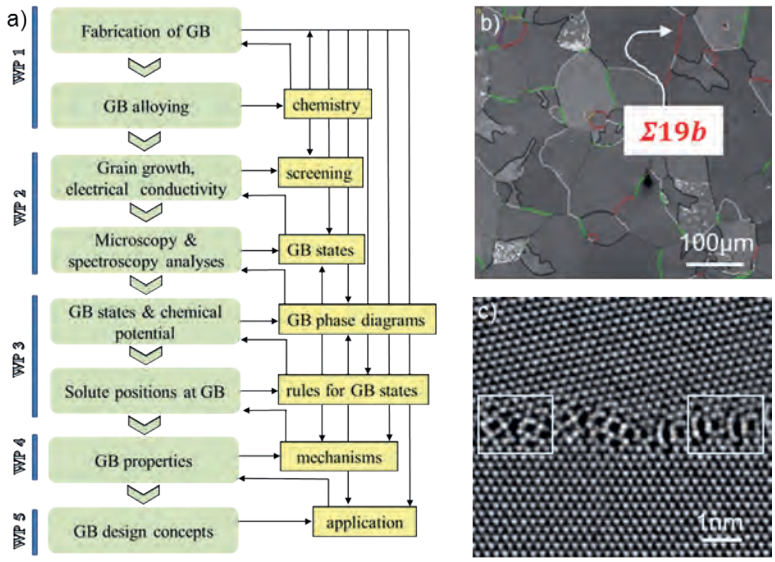


Fig. 1: *a) Overview on the work packages (WP) and their tasks within GB-CORRELATE. b) Oligocrystalline Cu film where different grain boundary misorientations are indicated by different colours; red lines mark ∑19b grain boundaries. (c) Atomic resolved scanning transmission electron micrograph of a ∑19b grain boundary with two different structural units (white boxes). Microscopy images are courtesy of T. Meiners.*

In addition to the structural exploration of grain boundaries, *in situ* micromechanical experiments will be performed to interlink grain boundary phases and mechanical properties. Grain growth experiments coupled with microscopy techniques such as electron backscatter diffraction and scanning electron microscopy will allow extracting changes in mobility of individual grain boundaries due to phase transitions in a high throughput approach. The project aims to predict and resolve grain boundary phase transitions, to establish guidelines for them, to correlate them with property changes and finally to provide design criteria for grain boundary engineering.

phase transitions. Cu, Al, and Cu- or Al-based alloys are employed as face-centred-cubic prototype materials with different stacking fault energies and melting temperatures. Growing oligocrystalline and strongly (111) textured polycrystalline thin films introduces further defects like triple lines but still provides access to the atomistic grain boundary structure due to a common zone axis for atomic resolved electron microscopy studies of neighbouring grains. The strategy of GB-CORRELATE is summarized in Fig. 1a, while Fig. 1b provides a first example of grain boundaries in an oligocrystalline Cu film, where two different grain boundary structures within a ∑19b grain boundary are resolved (Fig. 1c).

ERC Consolidator Grant: SHINE: How to See Hydrogen Atoms in Materials

B. Gault

Department of Microstructure Physics and Alloy Design (MA)

Despite being ubiquitous in nature, observing hydrogen (H) in matter is a formidable challenge. Hydrogen is elusive to scientific scrutiny like no other element, because of its low weight and extreme mobility. There is no doubt that H is a prime candidate for enabling future low-carbon emission manufacturing and power supply chains, but, as known for over 120 years, H is also at the origin of a strong and often sudden embrittlement phenomenon which has resulted in many catastrophic failures of engineering alloys and structures. Guided by promising preliminary results obtained on Ti-based alloys, summarised in Fig. 1, I decided to pursue a project targeting quantifying and locating H within materials. These results were the first obtained at the MPIE, where we could demonstrate the capacity of atom probe tomography (APT) to characterise stable hydrides, which are thought to be responsible for brittle fracture of a range of Ti-based alloys [1]. This Consolidator project funded by the European Research Council (ERC) is called SHINE as an abbreviation for 'Seeing Hydrogen IN mEtals'. Here, I ambition to exploit the unique facilities developed in the Atom Probe Tomography group at the MPIE to perform direct imaging and quantification of H atoms in candidate metallic alloys and materials for H-storage. The infrastructure of the Laplace Project allows for the preparation (see p. 34), electrochemical and, soon, gaseous charging of hydrogen or deuterium under highly controlled conditions. Following the transfer of the specimen under ultra-high vacuum and cryogenic conditions [1], these specimens can be investigated. Atom probe tomography will be the principal method of a correlative microscopy and spectroscopy approach to investigate materials where precise knowledge of the distribution of H is crucial. Informed by experimental data, modelling and simulations will provide a mechanistic understanding of the behaviour of H in materials.

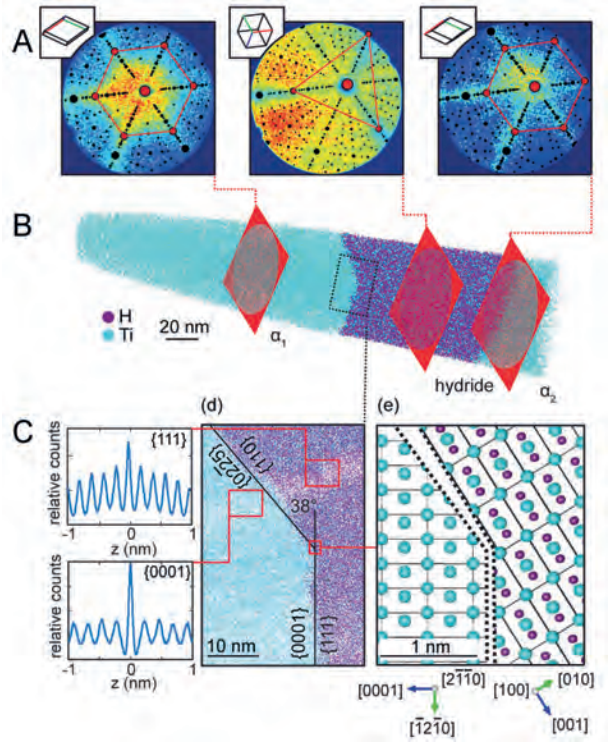


Fig. 1: *Atom probe crystallography analysis of a hydride precipitated at an α low-angle grain boundary in Ti-2Fe: (a) patterns formed on the detector during the analysis exhibiting the typical symmetries from the local crystalline phase highlighted by the superimposed stereograms; (b) APT reconstruction and (c) spatial distribution maps revealing the presence of atomic planes in the tomographic reconstruction near the α-Ti/hydride interface shown in (d); (e) model of the faceted α/hydride interface [1].*

Over the five years of the project, which started in February 2018, three PhD students and two post-doctoral scientists will work alongside the PI. This research is pursued in close collaboration with the department of Computational Materials Design, where two of the early-stage researchers are expected to be based. The expertise of the Interface Chemistry and Surface Engineering department will also come to support the development of novel hardware for gas-charging and UHV processing of metallic alloys. New data-processing methods, including possibly exploiting machine-learning approaches, will be established to maximise data quality.

The results from SHINE will be three-dimensional hydrogen mapping at the near-atomic scale that will provide new insights of the behaviour of H in the complex and dynamic microstructures of engineering materials. By relating this fundamental knowledge with observed physical properties, we will enable unprecedented precision in the prediction of material behaviour, and therefore pave the way for new materials design and manufacturing strategies to enhance their performance and durability. All these applications have relevance to a 'low-carbon-emission economy' that humanity must develop in the 21st century.

References

1. Chang, Y.; Breen, A.J.; Tarzimoghadam, Z.; Kürnsteiner, P.; Gardner, H.; Ackerman, A.; Radecka, A.; Bagot, P.A.J.; Lu, W.; Li, T.; Jägle, E.A.; Herbig, M.; Stephenson, L.T.; Moody, M.P.; Rugg, D.; Dye, D.; Ponge, D.; Raabe, D.; Gault, B.: Acta Mater 150 (2018) 273.

2. Stephenson, L.T.; Szczepaniak, A.; Mouton, I.; Rusitzka, K.; Breen, A.J.; Tezins, U.; Sturm, A.; Vogel, D.; Chang, Y.; Kontis, P.; Rosenthal, A.; Shepard, J.D.; Maier, I.; Kelly, T.F.; Raabe, D.; Gault, B.: doi.org/10.1371/journal.pone.0209211

ERC Starting Grant: TIME-BRIDGE: Time-Scale Bridging Potentials for Realistic Molecular Dynamics Simulations

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 aim of TIME-BRIDGE is to design novel methodologies that enable a more realistic atomistic simulation of materials. The challenge of present-day atomistic simulation techniques is to model 1) long time scales, 2) large system sizes with high accuracy and 3) to connect faithfully to experimental measurements. The general strategy of TIME-BRIDGE is therefore building upon three main pillars that focus on tackling these challenges under a common framework with steady exchange of ideas and expertise. A very good scientific progress was achieved during the first half of the funding period as evidenced by 20 publications in renowened journals and by many (>30) dissemination events on national and international level.

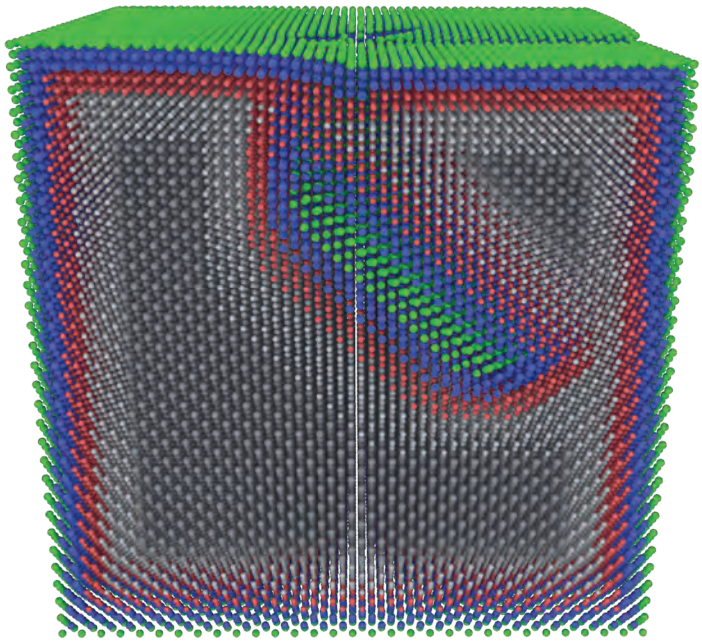


Fig. 1: *Partially indented nanopillar with an activated dislocation. The dark gray atoms are described by continuum theory (finite element method), blue and green atoms are treated by molecular dynamics and the other colours indicate buffer atoms.*

For example, we have investigated the possibility to use a new hyperdynamics potential (based on Smooth Overlap of Atomic Positions (SOAP)) for accelerating molecular dynamics simulations (A. Nematollahi). Our results show that this potential is able to differentiate between transition and equilibrium states for various types of simulations. Further, we have developed a new technique that bridges from the nano- to meso-scale by coupling atomistic calculations to continuum models (L. Huber). To achieve this, we use a state-of-the-art force-based coupling method between molecular mechanics (MM) and linear elasticity represented using the finite element method (FEM). We have now extended this technique to be fully adaptive in MM and FEM domains, even at finite temperatures. This adaptivity allows the MM domain to remain small, tracking defects which require atomistic resolution even as they are migrating through the system (see fig. 1). Within TIME-BRIDGE also small scale *in situ* compression experiments on nanopillars are performed to understand the size scaling of rare events such as dislocation motion and interaction with grain boundaries (N. Peter in collaboration with C. Kirchlechner from the SN department).

The TIME-BRIDGE project has a horizon of five years. Over this period, the funding covers three post docs, and a senior scientist.

ERC Starting Grant: CSI.interface: A Molecular Interface Science Approach: Decoding Single Molecular Reactions and Interactions at Dynamic Solid/Liquid Interfaces

M. Valtiner

Now at: Group Applied Interface Physics, Institute of Applied Physics, Vienna University of Technology

In 2016 Dr. M. Valtiner was awarded the prestigious ERC Starting Grant for his project: „CSI.interface - A molecular interface science approach: Decoding single molecular reactions and interactions at dynamic solid/liquid interfaces“.

Generating a detailed molecular understanding of complex, simultaneous interactions at reactive/dynamic solid/fluid interfaces is one of the biggest contemporary scientific challenges across disciplines. Whether it is during corrosive coating degradation, in biological adhesion, or during adaptive interfacial redox-cycle feedback in strongly adhesive seawater organisms: It is a large number of similar or dissimilar molecule/molecule, molecule/surface and competing interactions with ions/water that mediate complex macroscopic properties at crowded solid/liquid interfaces. How do single molecular interactions at dynamic, reactive or steady state interfaces translate into a macroscopically observable outcome? A truly molecular-level understanding of the complex length scale and chemistry dependent nonlinearities at interactive and reactive solid/liquid interfaces in 2D, let alone in 3D is missing.

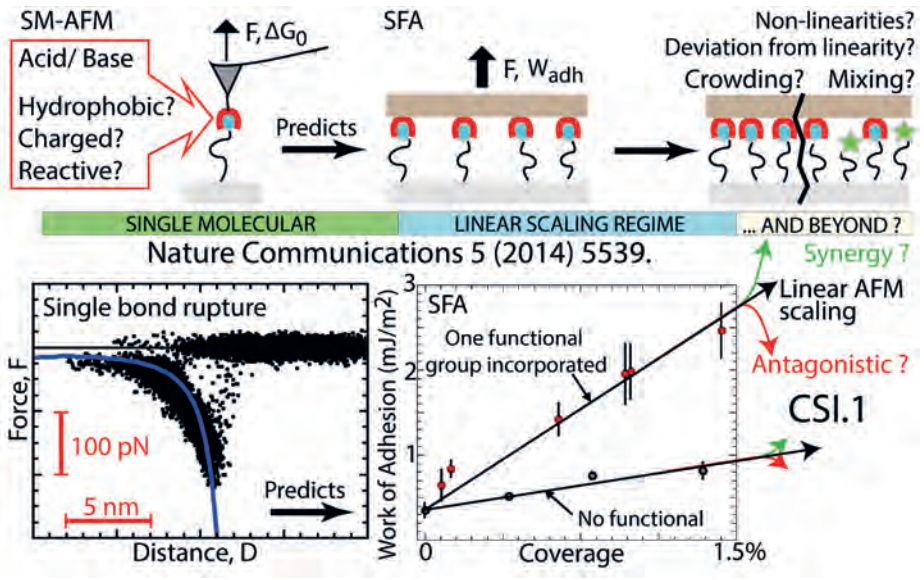


Fig. 1: *Bottom: The team of M. Valtiner showed how work of adhesion (W_{adh}) can be directly predicted from SM-AFM in a linear scaling regime. Top: In this ERC project this work will extend this to interfacial crowding, molecular mixing including the role of water, ions as well as interfacial reactions at all levels (single to integrating large numbers of interacting molecules).*

With the CSI.interface approach a predictive molecular insight into this complexity will be provided. The group of Dr. Valtiner will develop a comprehensive approach and tools in order to predict macroscopic properties/observables - such as interfacial adhesion or steady-state dynamics of redox cycles - based on single molecule experiments.

Combining Atomic Force Microscopy (AFM) based single molecule force spectroscopy and macroscopic Surface Forces Apparatus (SFA) experiments CSI.interface will (1) derive rules for describing nonlinearities observed in complex, crowded (water and ions) and chemically diverse adhesive solid/liquid interfaces; (2) uniquely characterize all relevant kinetic parameters (interaction free energy and transition states) of electrochemical and adhesive reactions/interactions of single molecules at chemically defined surfaces as well as electrified single crystal surfaces and step edges. Complementary, (3) a novel molecular force apparatus will be built in order to measure single-molecule steady-state dynamics of both redox cycles as well as binding unbinding cycles of specific interactions, and how these react to environmental triggers. CSI.interface goes well beyond present applications of AFM and SFA and has the long-term potential to revolutionize the understanding of interfacial interaction under steady-state, responsive and dynamic conditions. This work will pave the road for knowledge based designing of next-generation technologies in gluing, coating, bio-adhesion, materials design and much beyond.

M. Valtiner was offered a position at Vienna University of Technology and is now full professor, heading the Group “Applied Interface Physics“.

ERC Co-Investigator Grant: SMARTMET: Utilizing Phase Instability to Design Alloys with Enhanced Mechanical Stability

C.C. Tasan [1], J. Zhang [1], D. Raabe [1], B. Grabowski [2], L. Huang [2], J. Neugebauer [2]

[1] Department of Microstructure Physics and Alloy Design (MA)
[2] Department of Computational Materials Design (CM)

Prof. D. Raabe and Prof. J. Neugebauer were awarded an ERC 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) and worked on during the period 2012 to 2017.

The overarching aim of SMARTMET was the design of advanced metallic alloys with adaptive nanostructures by utilizing mechanically unstable phases. More specifically, the SMARTMET target was to break the well-known inverse strength-ductility challenge, which puts an apparent limit to the mechanical optimization of advanced engineering alloys. The solution approach we have introduced in SMARTMET was to develop new alloy design strategies based on the counter-intuitive approach of utilizing phase instability rather than phase stability. This target was pursued by the joint use of experiments and atomic-scale theoretical tools. The goals of SMARTMET have been reached leading to significant achievements in advanced alloys as evidenced by the more than 50 related publications in high impact journals.

As a representative example Fig. 1 shows the results achieved by bringing the SMARTMET idea into the field of high entropy alloys (HEAs). While over the past decade, these HEAs, also referred to as compositionally complex alloys or multi-principal element alloys, have drawn attention in the context of entropy driven stabilization of a specific phase, we changed this approach and rendered them more instable. Taking such a mechanistic perspective we developed entirely new HEA variants such as TWIP, TRIP, and dual phase HEAs with excellent mechanical properties (Fig. 1).

For this purpose several of the new mechanistically designed HE alloy variants violated some of the original HEA rules, such as using less than 5 alloying elements and the use of minority alloy ingredients below 5 %. Allowing for such compositional deviations from the original HEA rules was justified by three aspects: theory showed that the mixing entropy curves assume a shallow shape so that deviations from equimolar compositions do not alter the entropy configuration substantially. Second, the necessity for compositional tuning of a specific magnitude of the stacking fault energy or similar thermodynamic key quantity is more important than yielding maximum entropy. Third, when striving for excellent mechanical properties, avoiding brittle intermetallic phases is much more important than obeying static alloy design rules.

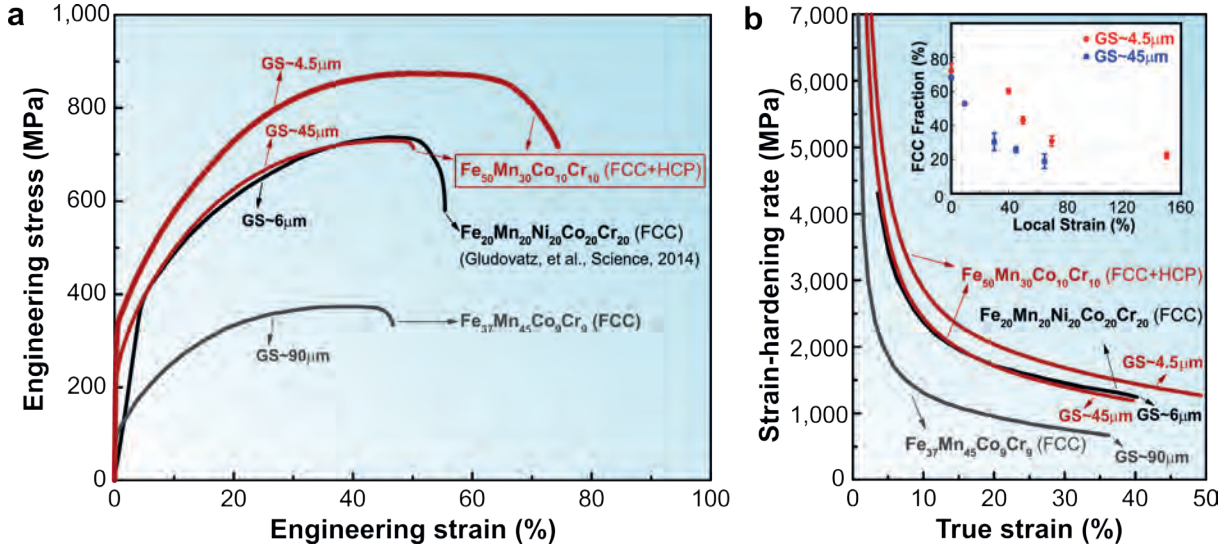


Fig. 1: *Mechanical behaviour of the TRIP-DP-HEAs compared to various single-phase HEAs. Ref: Nature 534 (2016) 227.*

Heinz Maier-Leibnitz Award

C. Kirchlechner

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

Since 1977, the German Research Foundation (DFG) annually awards about 10 early career scientists across various disciplines with the prestigious Heinz Maier-Leibnitz Award. The prize is named after the former president of the DFG and aimed for honoring young researchers and supporting them to pursue their scientific career. In May 2017, Dr. Christoph Kirchlechner, head of the Nano- and Micromechanics group in the SN department of the MPIE was the first time awardee coming from the MPIE in Düsseldorf. The award was presented by the federal minister of education and research, Prof. Johanna Wanka, and the president of the DFG, Prof. Peter Strohschneider in Berlin.

Dr. Christoph Kirchlechner is focusing on the mechanical properties of materials at the micron and submicron scale with special interests on interface fracture and plasticity. His toolbox comprises several electron and X-ray based in situ techniques, such as X-ray Laue microdiffraction (μLaue), which he conducts at the European Synchrotron Radiation Facility (ESRF, Grenoble, France). During the last five years Dr. Kirchlechner has used this technique to shed light on fundamentals of dislocation-grain boundary slip transfer mechanisms, a process of utmost importance for the manufacture and application of all engineering materials. Dr. Kirchlechner and his team could show that – in a certain size regime – the prominent Hall-Petch type hardening is a grain size and – at least at low strains – not a grain boundary effect. Furthermore, they were able to measure the dislocation transmission stress through grain boundaries [1], its strain rate sensitivity [2] and activation volume (submitted to Acta Materialia) for the first time.

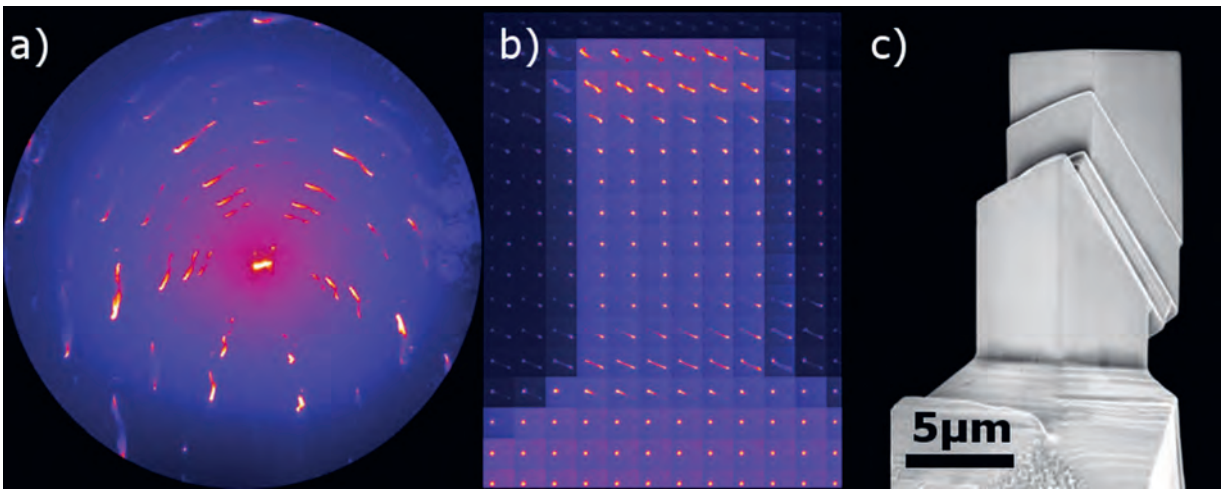


Fig. 1: *(a) A full Laue microdiffraction pattern recorded with a 1μm sized synchrotron beam on deformed tungsten. (b) A mosaic composition of Laue patterns recorded across the copper pillar presented in (c).*

References

1. Malyar, N.V.; Micha, J.S.; Dehm, G.; Kirchlechner, C.: Acta Mater. 129 (2017) 91.
2. Malyar, N.V.; Dehm, G.; Kirchlechner, C.: Scr. Mater. 138 (2017) 88.

NWO Grant: How to Mix the Perfect High-Entropy Alloy Cocktail

F. Körmann

Department of Computational Materials Design (CM)

The Dutch Research Organisation (NWO) supports the research of Dr. Fritz Körmann, project group leader in the CM department and researcher at the Delft University of Technology in the Netherlands with 800.000 euros for five years. The funding enables researchers to establish a research group in the Netherlands and to develop their own line of research. The research topic of Körmann is complex alloys including the new materials class of High-Entropy Alloys (HEAs). Metallic alloys constitute one of the oldest developments of sciences for thousands of years. It is therefore surprising when a new class of metallic alloys is discovered. High Entropy Alloys (HEAs) are such a class and have received great attention recently in terms of the underlying physics responsible for their formation as well as unusual physical and materials properties. HEAs reveal auspicious magnetic and outstanding mechanical properties, making them candidates for next-generation of technological applications. Whereas traditional alloys usually contain 1-2 main components, HEAs are comprised of 4-6 elements in high or even equal concentrations and crystalize into surprisingly simple lattice structures with randomly dispersed atomic species. Prototype examples are CrMnFeCoNi base HEAs. So far more than 30 different elements have been used resulting into more than hundreds of reported HEAs. Still this is only a marginal fraction of the immense number of possible combinations, leaving an overwhelming number of alloys with presumably superior properties unexplored. Further, basic mechanisms such as the relation of strength and chemical complexity are still poorly understood.

In this project we will address these issues by developing a fully parameter-free computational framework to predict materials properties of multi-component alloys. High-throughput first-principles computations will be performed to cope with the large configuration space, focusing in particular on stability and intrinsic materials properties, such as the stacking fault energy and local atomic fluctuations, which can be linked to macroscopic mechanical properties and mechanisms (e.g. twinning induced plasticity). As envisioned by the Materials Genome Initiative, this work represents a computational guide for the discovery and design of materials with specifically desired properties. This will greatly facilitate experimental efforts by narrowing down the large number of possible materials to well-defined promising candidate alloys, and thus greatly accelerate the exploration of hitherto unexplored materials with extreme properties for technological applications.

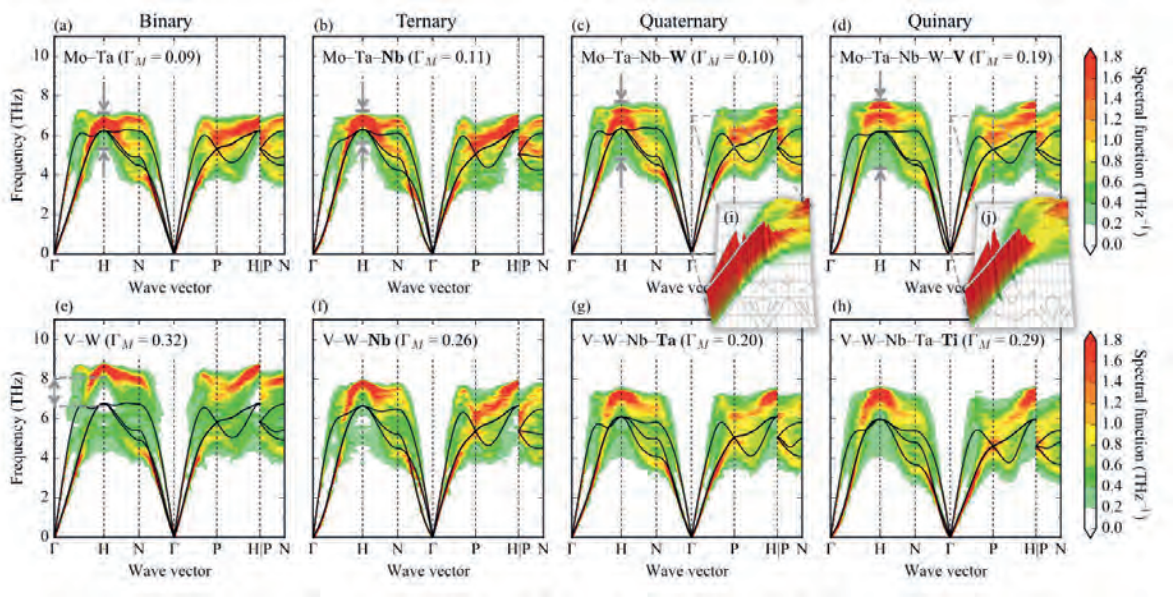


Fig. 1: *Broadening of phonon spectra with increasing number of constituent elements: From binaries to 5-component high entropy alloys. The added element for each alloy from left to right is shown in bold. Phonon spectra derived by employing averaged force constants and masses are shown for comparison as black solid lines. Insets (e) and (f) provide 3D representations of the long-wavelength phonon spectra for MoTaNbW and MoTaNbWV. Further details given in [1].*

References

1. Körmann, F.; Ikeda, Y.; Grabowski, B.; Sluiter, M.H.F.: npj Comput Mater 3 (2017) 36.

Heisenberg Fellowship

R. D. Kamachali

Department of Computational Materials Design (CM)
Department of Microstructure Physics and Alloy Design (MA)

The Heisenberg Programme by the German Research Foundation (DFG) is dedicated to promote young, highly-qualified researchers heading for professorship. It is one of the most renowned funding schemes of the DFG, named after German physicist Werner Heisenberg. The fellowship is supporting the awarded scientist in conducting independent researches for three years and can be extended to another two years. In October 2017, Dr. Reza Darvishi Kamachali was awarded the prestigious Heisenberg Fellowship to pursue his research on "Studying and Design of Chemo-mechanically Heterogeneous Microstructures Using Full-field and Mean-field Modelling".

The current fellowship is hosted by the departments of Computational Materials Design and Microstructure Physics and Alloy Design where scale-bridging studies across atomistic simulations and precise experimentation and characterization is highly developed. By combining mesoscale full-field simulations with mean-field modelling, Dr. Kamachali aims to further bridge the researches at the MPIE on the microstructure level. The strategy of the research is portrayed in the figure above. While mesoscale full-field simulations enable analyzing virtual microstructure evolution in full details, mean-field modelling allows the discovering of applied interrelationships between bulk material properties and the material's microstructure. Complex steels, aluminum alloys and high entropy alloys are potential materials of the research.

Dr. Kamachali is an expert in thermodynamics and kinetics of microstructure evolution. He conducted several researches on grain growth and nano-grain growth, recrystallization and precipitation in aluminum alloys. Dr. Kamachali is a principle investigator in the Priority Programme SPP1713 "Strong coupling of thermo-chemical and thermo-mechanical states in applied materials" where he developed a concept of chemo-mechanical coupling applicable in solid-state microstructure evolution [1,2]. He previously was leading the research group of Solid-solid Interface Kinetics at the Interdisciplinary Centre for Advanced Material Simulation, Ruhr-Universität Bochum.

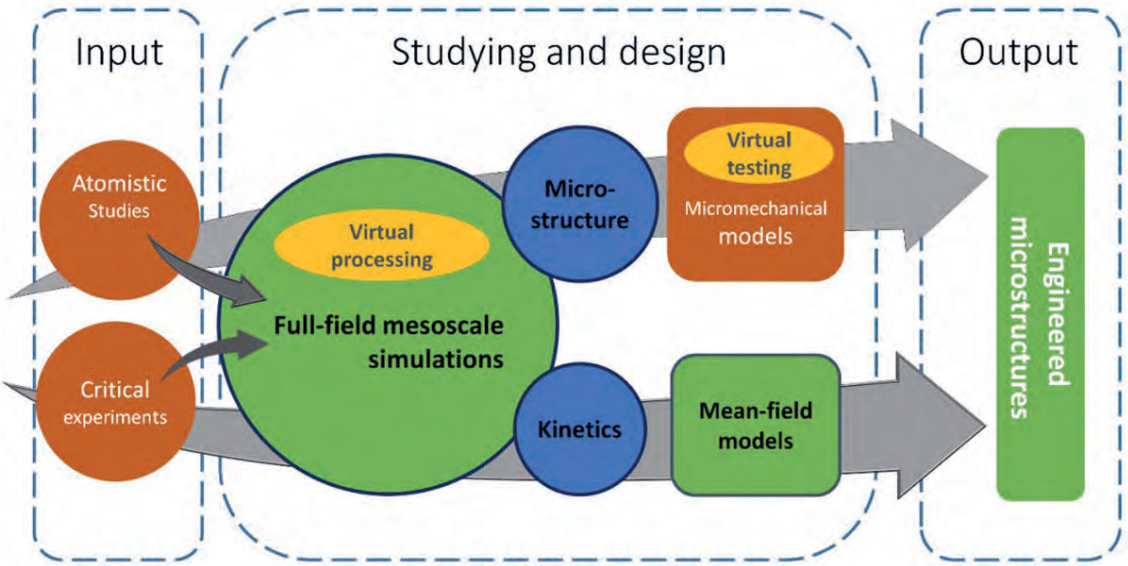


Fig. 1: *A strategy of a computationally-guided microstructure design.*

References

1. Kamachali, R.D.; Schwarze, C: Comp Mat Sci 130 (2017) 292.
2. Schwarze, C.; Gupta, A.; Hickel, T.; Kamachali, R.D.: Phys Rev B 95 (2017) 174101.

Research Funding

K. Hübel

Research Coordination Office

Professional Support in Research Funding

In the past six years 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 career prospectives of our junior scientists. She gave advice on all kinds of funding from individual (e.g. Humboldt Research Fellowship for Postdoctoral Researchers, Marie Skłodowska-Curie Individual Fellowship or 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).

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.

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

Interview Training

Especially applications for group leader funding often include interviews. Those are intensively practiced with participation of the Research Coordinator, the Public Relations Officer (see p. 83) and of course experienced scientific colleagues.

Project Management

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

Career Development

A special focus was set on the career development of young scientists. This included not only finding the right funding scheme that fits to their scientific profiles but also strategic advice for their career development. Furthermore Katja Hübel yearly gives a talk “Competitive Proposal Writing“ in order to qualify the researcher for writing high-quality, well-structured proposals.

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. Baptiste Gault was awarded with the Consolidator Grant of the European Research Council for his research about hydrogen mapping. Advanced characterization techniques such as atom probe tomography as seen in the photo, will be used.*

Selected Research Funding (2013 – 2018)

Prof. Gerhard Dehm received an ERC Advanced Grant for the project “GB-CORRELATE - Correlating the State and Properties of Grain Boundaries”, Mar 2018.

Dr. Baptiste Gault received an ERC Consolidator Grant for the project “SHINE - Seeing Hydrogen in Matter”, Feb 2018.

Dr. Blazej Grabowski received an ERC Starting Grant of the European Research Council Jul 2015.

Dr. Michael Herbig received 1.468 Mio € funding by the BMBF for his junior research group “Materials Science of Mechanical Contacts”, Sep 2016.

Dr. Cem Tasan former leader of the group “Adaptive Structural Materials” (until Dec 2015) won the Freigeist Fellowship of the Volkswagen Foundation, Jun 2015.

Dr. Markus Valtiner received an ERC Starting Grant for the project “CSI.interface - A molecular interface science approach: Decoding single molecular reactions and interactions at dynamic solid/liquid interfaces”, Dec 2015.

Promotion of Young Scientists

K. Hübel

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

Scientific Honours and Apprenticeship Awards

Our excellent junior researchers are rewarded with prestigious scientific honours. In 2017 the MPIE reached the 3rd best position in the Humboldt-Ranking among all non-university research organizations in Germany. In the last six years, our young scientists got a total of 115 prizes, awards and prestigious grants like ERC Starting and Consolidator Grants or a Freigeist Fellowship. The great success of our researchers is also due to the expert support in writing competitive proposals of our Research Coordinator Dr. Katja Hübel (see p. 77). Not only our scientists get honoured, the MPIE is also successfully educating apprentices. From 2013 - 2018 our trainees won 3 times the Apprentice Prize of the IHK (Chamber of Commerce), Düsseldorf and also 3 times the Apprentice Prize of the Max Planck Society.

Scientific Output

Our training, coaching and mentoring efforts result in a high scientific output and are also reflected by the career development of our youngsters. In the last six years junior scientists were author on 4 Nature papers, 12 Nature Communications and on 3 Science papers. Our young scientists are encouraged and financially supported to present their research results in international conferences, which is in addition supporting their networking activities and their recognition within the scientific community.

In the past 6 years, more than 10 of our junior scientists secured international professorship appointments at leading research institutes and universities like the Massachusetts Institute of Technology (MIT)/USA or the Imperial College London/UK. We support our senior postdoctoral researchers as well as our group leaders in doing their next career steps. The directors are sharing their expertise on selection committees for becoming university professor by training the interview situation with the respective researcher. Also applicants for the Max Planck Research Groups are prepared for the Selection Symposium. Prof. Christina Scheu is the "MPIE Female Science Career Mentor" supporting female senior postdoctoral researchers as well as young group leaders on their way to become a professor (see p. 81).

Teaching and Supervision

Teaching expertise and supervision activities are important prerequisites for an academic career. We encourage our researchers to do both which is facilitated by our connections to the Ruhr-Universität Bochum and the RWTH Aachen University.

From Researchers for Researchers

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

Public Relations

To attract also the next generation of scientists (pupils and students) the Institute's Public Relations Officer Yasmin Ahmed Salem is organizing a variety of events (see p. 83). The Institute participates in the Researchers' Night in Düsseldorf, we open our laboratories yearly during the Girls' Day, the MPIE is organizing Kids' Labs and we are participating in the Science Days of the Theodor-Fliedner grammar school. Researchers get a PR Coaching if they are invited to give an interview or if a short TV-movie is made to present their research. Also schools are coming to visit our Institute on a regular basis and the departments host pupils for 1-2 weeks of internships. Additionally, summer interns for students are provided by the institute and including a well-established summer internship programme with the students of materials science from the University of Cambridge (UK).

Applications of Excellent Researchers

To get excellent junior researchers, the MPIE has a doctoral progamme the „International Max Planck Research School for Interface Controlled Materials for Energy Conversion (IMPRS-SurMat)" (see p. 51). Postdoctoral researchers from abroad can apply for a PostDoc Scholarships (up to 10 years after PhD) or a Research Scholarships (more than 10 years after PhD). In all career stages speculative applications are possible.

International Office

S. H. Zwaka

Research Coordination Office

While the international researchers at the Institute were previously supported to different degrees by their respective departments and groups, in 2018 the board of directors of the MPIE decided to create an International Office at the Institute. By deciding to centralise and structure the support for the Institute's foreign scientists, the directors decided to follow the example of other Max Planck Institutes, research institutes and universities in Germany.

In September 2018, Sophia Helena Zwaka started working at the MPIE to set up an International Office. In order to create a fitting concept for the MPIE, during the first month, she talked to various parties at the Institute including the directors, assistants and secretaries of the directors as well as group leaders, PhD representatives and postdoctoral students.

The International Office aims at assisting the Institute's international scientists during their onboarding process and stay in Germany. This is to be achieved via personal support and the timely provision of relevant information via email, events and the Institute's intranet. Advice and information are offered regarding all aspects of everyday life in Germany including German bureaucracy, housing and family life.

During the first months after the official presentation of the International Office amongst the staff of the MPIE, Sophia Helena Zwaka has provided support in the following aspects: Providing information on visa processes and supporting the scientists in obtaining the adequate residence permit. Finding housing on short notice, which has been facilitated by the decision of the International Office to create a list of landlords who rent out their apartments on a regular basis. Furthermore, information on matters regarding health insurance and the ccupational pension scheme were provided upon request. Zwaka has also supported researchers residing in Germany with their families, by helping with visa and employment matters for spouses, as well as providing information for the application for public child benefit (Kindergeld) and guidance for researchers planning to have a child in Germany.

The majority of researchers who have sought support from the International Office came from the following countries: China, India and Iran.

In order to offer centrally available information, the International Office has created a checklist as an overview on the first steps in Germany. It aims at making relevant information available to every researcher by updating the Institute's intranet and to organise regular events. The organisation of a Welcome Coffee aims at improving interdepartmental communication and at better integrating new international employees by answering any lingering question on administrative processes. A second event aims at explaining the administrative procedures of the Institute to the foreign scientists. To facilitate the comprehension of administrative processes, the International Office works closely together with the HR department. Zwaka aims to facilitate communication between administration and researchers, by explaining the Institute's operating agreements and translating for the administration.

The International Office also plans to coordinate the provision of German classes in 2019, to organise information sessions on topics requested by the scientist and to create a buddy system, were every new foreign employee at the Institute has a buddy - a more experienced employee - in order to offer advice and support in the first weeks at the MPIE.

PhD Representatives

V. G. Arigela, J. B. Molin, R. S. Varanasi, A. Hariharan, S. Surendralal, L. Sreekala, P. Beley, M. Pander, J. S. M. Ochoa

The PhD representatives are the interdepartmental representation of the PhD students at the Max-Planck-Institut für Eisenforschung (MPIE). Annually the MPIE' s PhD students elect one external representative and eight internal representatives (two per department). We are responsible for maintaining and promoting the interdisciplinary cooperation among the PhD students at the MPIE but also in close contact with the other PhD students of the Max Planck Society by engaging within the PhDnet, a platform for exchange among doctoral students of all Max Planck Institutes. Moreover, we provide our fellow colleagues at the MPIE with up-to-date information about stipend and contract possibilities, insurance information and other upcoming and important topics for a better research and living experience. And we exchange important topics with the management of the Institute. We also serve as a bridge to the newly formed N^2, a common platform for doctoral students from Max Planck institutes, Leibnitz and the Helmholtz institutes.

Fig. 1: *During the visit to Dillinger Hütte, a steel manufacturer in Saarland, Germany. The PhD students are standing next to the longest steel slab ever produced by the company.*

The PhD representatives organize soft skill courses for PhD students biannually covering topics such as scientific writing, presentation, and leadership skills. Additionally, career talks are organized to give PhD students and postdoctoral fellows a platform to network with industry and gain insight into job opportunities from different industrial sectors. During the 2016-18 period, career talks with companies like Tata Steel and Dillinger received much positive feedback. The career talks are followed by visits to the respective manufacturing plants. Moreover, we coordinate with leading international research institutions to organize career events for students who opt to stay in academia. 2018 the National Institute of Materials Science, Japan, visited the MPIE and introduced their center and job opportunities. The MPIE lecture series, which was launched in 2015 to bolster the knowledge transfer in the Institute, flourished well in the past years. The lectures are organized by the PhD representatives, recorded and made available online for the in-house researchers. Exemplary lectures were delivered covering a wide variety of topics such as how to write competitive research proposals or how to use and interpret data from electron backscatter diffraction. The lectures are given by experienced MPIE group leaders. We also coordinate with the doctoral programme of the Institute IMPRS- SurMat to extend the reach to partner institutes and universities. The PhD representatives play an active role in social events of the institute throughout the year, such as the annual Christmas party.

Equal Opportunities

S. Degner, C. Scheu

During the last three years the MPIE has taken several measures to reconcile the demands of family and career.

Firstly, we introduced the family-service of the Max Planck Society to our employees in 2016; the "pme-Familienservice" offers support in childcare, recruitment of daycare-nannies/ daycare-solutions, emergency-nanny in case of child´s sickness, Au-Pairs, private "Kindergarten"/ nursery, programmes during school holidays, backup-service, eldercare, supervision of children and elder persons in general (homecare) and a hotline. The Institute is regulary providing current information on different offers of the "pme-Familienservice" via Intranet as well as via flyers and e-mail.

Secondly, we worked on finding a daycare facility for children (so called KiTa) near the Institute to be able to reserve some places for employees having small children (≤ three years). The reason for that is the lack of KiTa-places in Düsseldorf - especially for the group under three years. Especially our researchers from abroad encounter a lot of problems to get such places for their children. Starting in summer 2017 with two so called "Belegplätze" (places reserved for the MPIE) we increased the number in 2018 to four due to the huge demand.

As some situations in life of our employees may be challenging, the Institute tries to meet the needs of these employees in regard of working time and place.

In spring 2018, Simone Degner was elected as Gender Equality Officer. As one of the first measures she plans a parent-child-room within the next year to improve the flexibility for our staff in difficult childcare-situations.

Fig. 1: *Since summer 2017, MPIE employees are able to access places at a private children daycare centre, a so-called KiTa.*

Since October 2018, Prof. Christina Scheu is our "MPIE Female Science Career Mentor". In her function she is supporting female senior postdoctoral researchers as well as young group leaders on their way to become a professor. She has long-standing expertise in coaching as she was already a mentor for female habilitation candidates at the Ludwig-Maximilians-University in Munich. Currently, she has six mentees, from which one got a research grant to establish her own group. Prior to the official start of the mentoring programme, Prof. Scheu was involved in coaching a female scientists who got promoted to a professor position abroad.

The Institute successfully nominated Dr. Olga Kasian for the "Sign Up! Career Building Programme For Excellent Female Post Docs in the Max Planck Society".

Alumni Networking

B. Kohlhaas

The MPIE has a long tradition: in over 100 years, thousands of scientists have done research at our Institute, thus establishing the basis for their careers or using it as a milestone for promising scientific careers both in academic research and in industry.

Fig. 1: *The President of the MPG Prof. M. Stratmann, giving a speech at the Milestone Anniversary (Katja Velmans, MPIE GmbH).*

Over the years, a considerable number of employees have worked in various areas and departments and have contributed to the Institute's scientific success. In order to promote a lively exchange among the former Institute members, our Alumni Network was founded in 2003 with currently 1000 members.

The close contact to our alumni as well as to our active employees, is more than just a tradition for us. It is part of our Institute's philosophy and culture: a motor for synergy between research and practice.

What does "Alumni Network" mean?

An alumni network is an association of alumni, originally consisting of former university students.

Today, the term commonly refers to people who have spent a certain part of their lives at a company, school or university and have reached the next steps of their own further education. Trainees, students, active and former employees belong to the target group of alumni networks.

Who are Alumni?

Anybody who wants to stay in contact, even after his or her time at the Institute, can register as alumni. All former employees of the Institute - whether scientifically active or not - are alumni. This includes visiting scientists who were at least six months at the Institute, trainees, undergraduates, graduate students, fellows and postdocs as well as retired colleagues.

What are the aims of the Alumni Network?

The network cultivates contacts 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 - across workspaces and generations, across national borders.

Keep in Touch

The alumni network helps 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.

The members of the alumni network are updated about news, events and actions of the MPIE by 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.

Fig. 2: *Networking at the 100 years anniversary (Katja Velmans, MPIE GmbH).*

In addition, one can inform about Alumni News and easily join the Network online https://alumni.mpie.de.

Alumni Meeting 2017 at the Milestone Anniversary

In 2017, we looked back over a history of 100 years of the Max-Planck-Institut für Eisenforschung GmbH. This gave us the opportunity to invite all of MPIE's former colleagues to a festive event. **On 6th of October** we celebrated our **Milestone Anniversary** with an official ceremony followed by a buffet and a party! More than 500 guests attended the successful event, including alumni and current MPIE employees.

The Institute in Public

Y. Ahmed Salem

Research Coordination Office

The MPIE's Public Relations (PR) are based on two main pillars: press relations and events. Some examples of the work between 2016-2018 are highlighted here.

Press Relations

The press relations of the MPIE consist of press releases, short news, articles and other printed and online publications, which are written or supervised by the PR Officer.

Throughout the years 2016-2018, the **press releases** issued new research projects with special funding, awards and important scientific publications. This way, our latest findings were illustrated to a broader public, revealing the importance and depth of our research. Press releases are send out to journalists, published on the MPIE website and in the idw, an online service offering latest news to a wider range of interested journalists and people from industry and academia.

Examples of press releases in the reporting time:

11 Oct 2016: Was Windkraftanlagen mit Bahnschienen gemeinsam haben (engl.: What wind turbines and railway tracks have in common) (Funding of the German Ministry for Education and Research to Dr. Michael Herbig)

1 Feb 2017: Gum metals pave the way for new applications (Publication in Nature Communications)

9 April 2018: Oxygen: A blessing and curse for nanostructured alloying (Publication in Nature Communications)

15 Aug 2018: Designing nanocrystals for more efficient optoelectronics (Publication in Nature Nanotechnology)

28 Nov 2018: How oxygen can enhance strength and ductility of high-entropy alloys (Publication in Nature)

News are scientific highlights that are presented on the website and are mainly about awards to our scientists or events taking place at or being organized by the MPIE. News appear more often and give an up to date insight on what is happening at the Institute.

Examples of news in the reporting time:

11 April 2016: Martin Friák wins Best Poster Award of the German Physical Society

10 Jul 2017: Enrico Pizzutilo at the 67th Lindau Nobel Laureate Meeting

20 Sep 2018: First Health Day at the Max-Planck-Institut für Eisenforschung

The **MPIE newsletter** appears twice a year. Since 2014, the newsletter focusses on one research topic per edition thus showing the interdepartmental collaboration and different approaches and aspects of one highlight. The topics are either chosen or suggested by the press officer and always contain participation of at least three departments. The newsletter is distributed to industrial partners, journalists and at events in and outside the MPIE. Between 2016 and 2018 the topics were: high entropy alloys, high temperature oxidation, grain boundaries, big data and intermetallics.

Each year all Max Planck Institutes contribute with one or two articles to the **Yearbook of the Max Planck Society**. Since 2017, the Max Planck Society asks the Institutes to hand in articles, which are generally understandable and deal with society's hot topics in order to reach the target group of politicians and decision makers. Thus, the MPIE's PR Officer got in charge of writing the article for the yearbook. The first article was about bone-like steels that prevent materials' fatigue. This article was based on a publication in the prestigeous journal "Science". The vice presidents of the Max Planck Society chose the MPIE's article to be printed in a special book that contains only 15 out of over 80 articles of the yearbook, and which is distributed to politicians showing the relevance of the research in the Max Planck Society.

Events

Promotion of Young Scientists

To encourage young people to enter research, the PR participates in and organizes various events for children, pupils and students. One example are the **kids' labs**, which started in the jubilee year of the Institute. The PR advertised the event via e-mail, newspaper, posters and on the MPIE's website and was overwhelmed by the positive feedback. In 2017, a kids' lab for five to eight years old children was

Fig. 1: *During the Girls' Day 2018 at the MPIE.*

organized giving them the chance to make small experiments on their own. In 2018, another kids' lab was organized during the Max Planck Day (see below). Three **pupils' universities** were organized and held by Dr. Stefan Zaefferer, group leader in the Department of Microstructure Physics and Alloy Design, two of them in 2017 as part of the 100 years jubilee programme and one in 2018 for a class of a primary school in Flingern, a region of Düsseldorf. The Institute also participates at the yearly nationwide **Girls' Days**, where girls have the chance to experience one day as an employee in a field where women are underrepresented. This day is mainly organized by the Research Coordinator (see p.77) and advertised by the PR Officer. Another activity to promote pupils are the **Science Days** of the nearby Theodor Fliedner grammar school. The school organizes yearly an event where companies and research institutes can present their work to the pupils in order to fascinate them for natural sciences. We participate in this programme since its upcoming in 2012 and the PR Officer chooses the doctoral students who present their work and trains them in giving a presentation to pupils. Students who participated in the reporting period were: 2016 - Waldemar Krieger and Julian Rechmann (Department of Interface Chemistry and Surface Engineering); 2017 – Michael Ackers (Department of Microstructure Physics and Alloy Design); 2018 – Caroline Fink (Department of Structure and Nano-Micromechanics of Materials). Groups of **students visit** the MPIE on a regular basis. Hereby the PR Officer organizes a day at the Institute, giving the students a chance to get an insight of the research and possibilities to do a masters or Ph.D. degree. Students and pupils are also welcome to do an **internship** at the MPIE. The internship applications either directly go to the scientists and are handled there, or they reach the Research Coordination Office and are distributed among the departments. Especially the pupils are often supervised by the trainers of the six apprenticeships.

Events for the Broad Public

KopfSalat is an event series taking place twice a year at the MPIE and inviting speakers from all fields of research to give generally understandable talks to a broad public. It is organized by the PR and intends to foster the popularity of the Institute in and around Düsseldorf. Having started in 2014 with around 70 visitors, KopfSalat now has a huge fan community with about 180 visitors per talk. Since 2018 the KopfSalat takes place in the newly build event facilities of hall 9 allowing such a huge audience. The event is announced through the PR mailing list, which in 2018 contained about 300 subscribers, in the local newspaper, different online and social media sites and through post cards that are distributed in the neighbourhood of the Institute, at the Heinrich Heine University Düsseldorf, at the Adult Education Centre Düsseldorf and the town libraries. Besides external speakers, also MPIE scientists gave talks:

Fig. 2: *The kids' lab during the Max Planck Day at the MPIE.*

Dr. Christoph Kirchlechner on modern microscopy using synchrotron facilities (2016), Prof. Dierk Raabe on materials research from the very beginnings till now (2017), and Dr. Christoph Freysoldt on big Data and artificial intelligence in materials science (2018).

The **Max Planck Day** took place for the first time on 14 September 2018. The Max Planck Society celebrated Max Planck's 160 years anniversary, his receipt of the Nobel Prize 100 years ago and the Max Planck Society's 70 years jubilee as the successor of the Kaiser Wilhelm Society. The MPIE's PR Officer was part of the organization task force and developed with colleagues from other Max Planck Institutes and the headquarters of the Max Planck Society the concept of this day. It was decided to offer events at

the Max Planck Institutes as well as central events in Munich and Berlin. Two events took place at the MPIE: a kids' lab for children between five and eight years with numerous interactive experiments and a KopfSalat-talk by Dr. Christoph Freysoldt (see above). At the same time a team of MPIE-scientists went to the central event in Munich, a science marketplace, to present their research about additive manufacturing using the exponates created at the Communicate Science Competition for the 100 years celebration (see below), and to take part at a science

Fig. 3: *Armin Laschet, Prime Minister of North Rhine-Westphalia, during his speech at the ceremonial act on 6th October 2017. Copyright: Katja Velmans, Max-Planck-Institut für Eisenforschung GmbH*

slam where researchers from different Max Planck Institutes explained their work in brief and entertaining talks. The same science slammer who took part at this science slam, Aniruddha Dutta, doctoral student in the Department of Microstructure Physics and Alloy Design, won in November 2018 the German Science Slam Championship.

Special Event: 100 Years Ceremony

In 2017, the Institute celebrated its 100 years anniversary with different events and publications starting from January 2017 and ending with a scientific symposium and a ceremonial act with over 550 guests in October. The preparations started one year before by defining the target groups and how to reach them. The MPIE's first image film, presented during the 100 years ceremony, was produced and is since then available on the website and used at various Institute's presentations. Moreover, an image brochure was created that presents the MPIE's research, its history and overall philosophy. The historic text was researched and written by a special agency, and is now available both on the website and as a flyer, both in German and English. To involve the broad public more into the 100 years celebrations, the PR organized almost monthly events as two pupils' universities, one kids' lab and four KopfSalat talks (see above). A "Communicate Science Competition" amongst the MPIE staff was organized, where scientists and technicians participated to present their research through (interactive) exhibits. These exhibits were presented during the ceremonial act and are now used whenever our research should be explained to a broad public.

Training and Consultant Service

The PR Officer is engaged in the training of scientists concerning presentation skills and event organization. Hereby the PR Officer works in close collaboration with the Research Coordinator to train scientists for their interviews that are part of the application procedure to receive research funding. Moreover, the PR Officer attends rehearsals for talks which are supposed to be given to a broad public or to non-researchers. Additionally, help is given in the organization of scientific conferences and events in the Institute, like the TEM-APT opening in November 2018, where the PR Officer was member of the organizing team. This close connection between PR and research gives mutual benefit as the PR Officer gets a closer insight into current research topics and the MPIE scientists get information on how to organize events with up to 200 people and what is needed for advertisement and marketing.