Scientists at the MPIE

High-performance nanostructured thin films – their fabrication, characterization and engineering are the object of the new research group of **Dr. Matteo Ghidelli**. He is head of the group "Thin Films & Nanostructured Materials" in the department "Structure and Nano-/Micromechanics of Materials". Ghidelli joined the MPIE in November 2018, after being postdoctoral researcher at the University of Roma Tre and the Politecnico di Milano in Italy.

"Working at the MPIE gives me the chance to collaborate easily with the in-house groups specialized on characterization and mechanical testing of sub-micrometer scale materials. Thus, it is possible to develop nanoarchitectured materials with advanced mechanical properties, which for example combine a high yield strength and high elasto-plastic deformations.", explains Ghidelli. His group mainly exploits physical vapour deposition (PVD) techniques including DC/RF magnetron sputtering, electron beam evaporation and molecular beam epitaxy to synthetize novel nanostructured thin films, while post-thermal annealing treatments are carried out to further tune film microstructure. Currently, four main projects are being followed:

- A project on the synthesis of novel thin film metallic glasses Zr-TL (late transition metal TL=Ni, Cu, Co) with controlled thickness, composition and morphology, while investigating the main mechanical properties and focusing on the nanometer scale deformation mechanisms.
- A project focused on dewetting of noble metal alloy and thin films aimed to investigate the nanoscale dewetting mechanisms as well as to reduce the amount of noble metal elements,. This is especially interesting for catalytic processes.
- A project dealing with the measurement of electrical properties of grain boundaries in metallic alloys and thin films. The impact of this project relies on an in-depth understanding of the grain boundaries structure with several applications in the field of metallurgy and novel engineered metallic materials and films.
- And a project on thin films high entropy alloy with the aim to synthetize nanostructured thin films with enhanced mechanical and thermal properties benefiting of the atomic complexity and engineered microstructure.

Possible applications of Ghidelli's research are mainly in the aerospace and energy industry, in microelectronics (i.e. micro-electro-mechanical systems, MEMS), as well as in the field of catalysis.

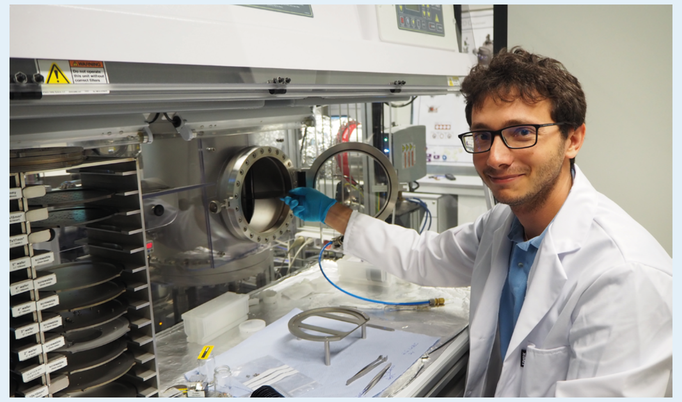

Fig. 1: *Dr. Matteo Ghidelli is head of the new group "Thin Films and Nanostructured Materials".*

Selected Publications:

1. J. Ast, M. Ghidelli, K. Durst, M. Goeken, M. Sebastiani, A.M. Korsunsky: Mater Design 173, (2019) 107762.
2. M. Ghidelli, M. Sebastiani, K. E. Johanns, G. M. Pharr: J Am Ceram Soc 100 (2017) 5731.
3. M. Ghidelli, H. Idrissi, S. Gravier, J.-J. Blandin, D. Schryvers, J.-P. Raskin, T. Pardoen: Acta Mater 131 (2017) 246.

Selected Publications

Computational Materials Design (CM):

J. Janssen, S. Surendralal, Y. Lysogorskiy, M. Todorova, T. Hickel, R. Drautz, J. Neugebauer: *pyiron - an integrated development environment for computational materials science.* Comp Mater Sci 163 (2019) 24.

T. Kostiuchenko, F. Körmann, J. Neugebauer, A. Shapeev: *Impact of lattice relaxations on phase transitions in a high-entropy alloy studied by machine-learning potentials.* npj Comp Mater 5 (2019) 55.

Interface Chemistry and Surface Engineering (GO):

L. Exbrayat, S. Salaluk, M. Uebel, R. Jenjob, B. Rameau, K. Koynov, K. Landfester, M. Rohwerder, D. Crespy: *Nanosensors for monitoring early stages of metallic corrosion.* ACS Appl Nano Mater, 2 (2) (2019) 812.

J. Peng, F. Moszner, J. Rechmann, D. Vogel, M. Palm, M. Rohwerder: *Influence of Al content and pre-oxidation on the aqueous corrosion resistance of binary Fe-Al alloys in sulphuric acid.* Corros Sci 149 (2019)123.

Microstructure Physics and Alloy Design (MA):

H. S. Oh, S. J. Kim, K. Odbadrakh, W. H. Ryu, K. N. Yoon, S. Mu, F. Körmann, Y. Ikeda, C. C. Tasan, D. Raabe, T. Egami, E. S. Park: *Engineering atomic-level complexity in high-entropy and complex concentrated alloys.* Nat Commun 10 (1) (2019) 2090.

Y. Bu, Z. Li, J. Liu, H. Wang, D. Raabe, W. Yang: *Nonbasal slip systems enable a strong and ductile hexagonal-close-packed high-entropy phase.* Phys Rev Let 122 (7) (2019) 075502.

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

J. Ast, M. Ghidelli, K. Durst, M. Göken, M. Sebastiani, A. M. Korsunsky: *A review of experimental approaches to fracture toughness evaluation at the micro-scale.* Mater Design 173 (2019) 107762.

S. W. Hieke, M. G. Willinger, Z. J. Wang, G. Richter, D. Chatain, G. Dehm, C. Scheu: *On pinning-depinning and microkink-flow in solid state dewetting: Insights by in-situ ESEM on Al thin films.* Acta Mater 165 (2019) 153.

Selected Talks

Computational Materials Design (CM):

J. Neugebauer, M. Todorova, B. Grabowski, T. Hickel: *Modelling structural materials in realistic environments by ab initio thermodynamics.* TMS 2019, San Antonio, USA, 10 – 14 Mar 2019

M. Todorova, S. Surendralal, J. Neugebauer: *First-principles approach to model electrochemical reactions at the solid-liquid interface.* DPG, Regensburg, Germany, 31 Mar – 5 Apr 2019

Interface Chemistry and Surface Engineering (GO):

M. Rohwerder: *Scanning Kelvin Probe based techniques for mapping hydrogen distribution in metals and their application for investigating hydrogen embrittlement.* Workshop "Hydrogen in Metals", St Anne's College, Oxford, UK, 15 – 18 Apr 2019

M. Rohwerder: *Hydrogen embrittlement, hydrogen traps, high sensitive detection of hydrogen with high spatial resolution, corrosion.* Dreiländerkorrosionstagung, Dechema-Haus, Frankfurt, Germany, 9 – 10 Apr 2019

Microstructure Physics and Alloy Design (MA):

D. Raabe: *The Bauerman Lecture Award 2019.* Named Lecture Award. Department of Materials, Imperial College London, Royal School of Mines, UK, 28 Feb 2019

D. Raabe, D. Ponge, A. Kwiatkowski da Silva, S. Makineni, S. Katnagallu, L. Stephenson, P. Kontis, X. Wu, C. Freysoldt, J. Neugebauer, B. Gault: *Chemistry at lattice defects probed at atomic scale.* The 53rd Annual Meeting of the Israel Society for Microscopy, Tel Aviv, Israel, 29 May 2019

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

C. Liebscher: *Exploration of interfacial transitions by correlating atomic scale microscopy with atomistic simulations.* TMS 2019, San Antonio, USA, 10 – 14 March 2019

J. B. Molin, L. Renversade, J. S. Micha, C. Kirchlechner: *Paving the way to unique, non-destructive 3D-microstructure properties by in situ Laue tomography.* DPG, Regensburg, Germany, 31 Mar – 5 Apr 2019

Selected Upcoming Events

13 Sep 2019: Night of Science 2019
The MPIE will present its research on additive manufacturing and nanomechanics during the upcoming Night of Science. The Night of Science is an event for the broad public taking place in the centre of Düsseldorf. Other participants will be among others the University of Düsseldorf, the University of Applied Sciences and the Forschungszentrum Jülich. The aim is to present modern research in a generally understandable way to attract especially potential students for the different fields of science.
http://www.nachtderwissenschaft-duesseldorf.de/

16 – 18 Sep 2019: Conference on "Theory of Complex Disorder in Materials 2019"
The workshop offers a platform to discuss recent trends of method development allowing the first-principles treatment of materials with complex disorder and excitations. This is especially relevant for complex and high-entropy alloys, magnetic materials and the usage of machine learning techniques. The conference takes place in Linköping, Sweden.
https://liu.se/en/research/tcdm2019

30 Sep – 4 Oct: Conference "Intermetallics 2019"
Materials based on intermetallic phases constitute a new class of materials, which encompasses alloys for structural and functional applications. The congress addresses all kinds and properties of intermetallics, and topics also include their role as strengthening phases in compositionally complex alloys or high entropy alloys. The conference takes place in Bad Staffelstein, Bavaria.
https://www.intermetallics-conference.de/

27 – 30 Oct: International Workshop on Advanced and *in situ* Microscopies of Functional Nanomaterials and Devices 2019
The workshop aims to provide a forum for researchers who are interested in applying advanced imaging and spectroscopy methods of electron microscopy to topical issues in materials science and engineering, in nanoscience, in soft matter research, in interface and surface science, and in biomaterials research. It will take place at the MPIE.
https://www.mpie.de/iamnano2019.html

7 Nov 2019: NRW-APT User Meeting
The meeting takes place for the 5th time and brings together scientists from North Rhine-Westphalia (NRW) dealing with atom probe tomography (APT) and correlating techniques. Aim of the meeting is to share knowledge concerning sample preparation, measurement conditions and data reconstruction & analysis.
https://www.mpie.de/4075335/5th-nrw-apt-user-meeting.html

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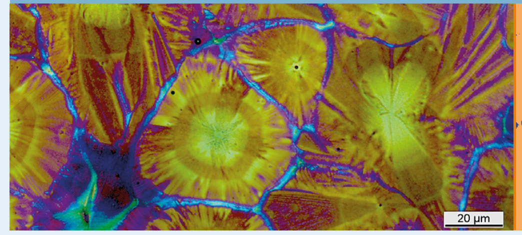
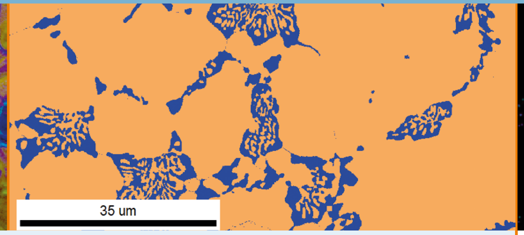
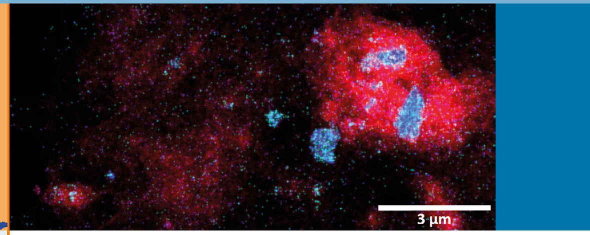






Damage Mechanisms in Complex Tribological Environments

From bearings and railroad tracks, to tooth or hip implants – the number of examples where materials are exposed to mechanical contact loads is as numerous as the number of materials used under such conditions. Failure in such applications is associated with enormous economic costs and can severely affect the well-being of people. Deepening our understanding of the fundamental damage and failure mechanisms associated with intense mechanical contacts and harsh environmental conditions is required to improve sustainability.

The contact between two solid bodies subjected to high forces under harsh environmental conditions and repetitive loading 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, the formation of tribolayers, or even corrosion or hydrogen embrittlement. These processes usually occur simultaneously while in service and cannot be tracked *in situ*. The analysis of such phenomena requires combined chemical and structural characterization down to the atomic scale.

One main focus of the research conducted in Dr. Michael Herbig's BMBF-funded group "Materials Science of Mechanical Contacts" is the investigation of white-etching-cracks (WECs), which are primarily known to cause failure in bearings and rails, but are in reality ubiquitous in high carbon steel applications subjected to intense mechanical contacts. Although this failure mode has been known for more than 100 years and causes enormous economic costs worldwide, so far neither is the mechanism understood nor are cost-efficient countermeasures available.

Our investigations showed that failure by WECs in rails and bearings involve fundamentally different conditions. In the case of rails, the comparison of experimental results with simulations revealed that each rail/wheel contact must be considered as an individual thermomechanical processing step of the rail. The high loads lead to high amounts of plastic deformation right below the contact patch in the rail. This is accompanied by frictional heating to peak temperatures exceeding 700°C that decay in a few milliseconds to ambient temperatures. The material changes accumulate during repeated loading of the rail, finally leading to a microstructurally altered, semi-brittle layer. Long fatigue cracks are generated when this layer fractures or partly delaminates [1].

In bearings, the situation is fundamentally different. The significantly lower contact forces and friction

EDITORIAL

Dear Colleagues and Friends of the MPIE,

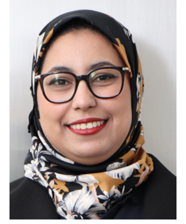
understanding the origin of material failure is essential for all applications where materials are exposed to mechanical contact and tribology. This newsletter edition therefore highlights some of our recent research on white etching cracks and fracture, phenomena, which decide about the life or death of such diverse devices as windmills or biomedical implants. Moreover, it presents our new group on thin films and nanostructured materials.

Please also have a look at the upcoming events and selected publications.

Enjoy reading,

Prof. Dr. Dierk Raabe, Chief Executive, MPIE

Awards and Achievements



Lamya Abdellaoui, doctoral student in the group "Nanoanalytics and Interfaces", received a Graduate Student Conference Registration Wavier for the "38th International Conference on Thermoelectrics" in Geyeongju, South Korea.

Dr. Michael Ashton, postdoctoral researcher from the United States, received a research scholarship of the Alexander von Humboldt Foundation and is now working in the group "Defect Chemistry and Spectroscopy".

Dr. Steffen Brinckmann, head of the group "Nanotribology", has successfully completed his habilitation at the Ruhr-Universität Bochum.

Till Freieck, former trainee and now materials tester at the MPIE, has been awarded as best trainee 2018 in the field of material testing by the Chamber of Industry and Commerce Düsseldorf.

Thomas Gänsler, doctoral student in the group "Nanoanalytics and Interfaces", won the second place at the image competition of the Royal Microscopical Society.

Dr. Matteo Ghidelli, head of the group "Thin Films & Nanostructured Materials", has been honoured with the "2018 MDPI Top Reviewer Award."

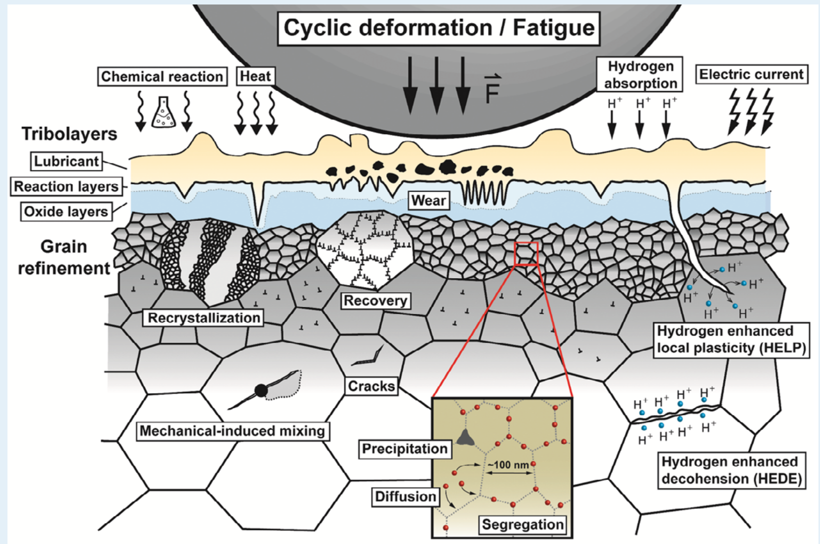


Fig. 1: *Microstructure alterations caused by intense mechanical/environmental contacts. Drawing by P.-Y. Tung.*

causes the highest shear stresses to be found below the surface. Here cracks initiate at 60-90°C at flaws and eventually propagate to the surface to cause the part failure. Our research activities now have revealed that this failure is accompanied by a so far unknown crack propagation mechanism. Cracks have been known to continuously extend until failure occurs and this holds true also for WECs. However, WECs show additionally to that a unique particularity: they move through the material, viz. they continuously change their position, leaving behind a severely plastically deformed area. This so-called white etching area consists of nanocrystalline ferrite along with the segregation of carbon on the grain boundary. As WECs in bearings initiate within the bulk, the corresponding fracture surfaces are not oxidized. When WECs get in contact during a compressive loading cycle, strong adhesive forces form and material can be transferred from one side of the fracture surface to the other. Many repetitive loading cycles lead to a macroscopic change in the crack position [2].

The research activities on steels are complemented by investigations on hip implants, where corrosion and wear debris at the contact zone between a titanium (Ti-6Al-4V) alloy stem and cobalt (Co-30Cr-4Mo) alloy head lead to adverse local tissue reactions. Here, the correlated use of the in-house high resolution techniques of transmission electron microscopy and atom probe tomography revealed that the passive film of the cobalt alloy can be torn off and be incorporated into the tribolayer of the titanium counterpart. The hence exposed fresh cobalt alloy surface corrodes, leading to the release of toxic Co into the human body [3].

Currently, cyclic mechanical contacts in liquid environments are often considered only in terms of stress fields, friction and lubrication flow. Our insights demonstrate the importance of material science aspects for the understanding of the related damage phenomena. Only when these additional factors are considered can the root causes of failure be determined and knowledge-based approaches to improve device reliability can be designed.

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MA authors: S. Balachandran, M. Herbig

Submicron Fracture Mechanics

Catastrophic fracture is omnipresent in structural and functional materials and material systems. Moreover, fracture is of utmost importance to increase the reliability of devices. Until now, the mechanisms of crack initiation and growth at the microstructure length scale are not thoroughly understood. Today, we therefore develop tools for measuring the fracture properties at the submicron scale in order to pursue the development of damage tolerant materials and material systems.

Materials either fail by plastic deformation, by fracture or by a combination of both. The fracture process can be subdivided into three stages: crack nucleation, crack growth and, finally, the catastrophic failure. Today, the multi-scale nature of failure is well-understood: While the crack nucleation typically occurs at the microstructural length scale, i.e. at the level of single grains, phases or interfaces, the subsequent crack growth and catastrophic failure typically involves several different length scales. Former experimental limitations in analyzing cracks on the order of a few hundred nanometers prevented a thorough understanding of the small length-scale impact on device reliability. However, recent advances in producing micron or even submicron sized structures by focused ion beam (FIB) milling open new horizons in analyzing the first failure stage: the crack nucleation and crack growth at the microstructural length scale.

For this purpose, we, the Nano- and Micromechanics as well as the Nanotribology groups of the department Structure and Nano-/ Micromechanics of Materials (SN) produce micro cantilevers with typical dimensions of 1x1x5 µm³ and 200 nm deep artificial notches by FIB machining. The notch root radius is of the order of 10 nm. Subsequently, the samples are loaded inside a scanning electron microscope (SEM) by an *in situ* straining rig – with forces on the order of 1 mN [1]. For brittle materials, the load increases until the local stress intensity at the artificial notch surpasses the material fracture toughness, i.e. critical strength against failure. The measured maximum force and the geometry factor (obtained from finite element simulations [2, 3]) are used to analyze the material dependent – but size independent – critical stress intensity factor, which is called fracture toughness. In case of semi-brittle materials, all plastic contributions to the energy dissipation have to be included. We are following the spirit of the internationally accepted ASTM[1] standards for macroscopic testing (e.g. using a micro *J* criteria) by developing experimental methods at the micrometer length scale.

The micro cantilever can be placed in any location in a real microstructure: single grain or phase, individual interfaces. Functional material systems are of special interest to the SN department: semiconductors (like silicon [4, 5] or silicon nitride), biological composites, materials for high temperature applications [6] and hard coatings. Using these micro fracture experiments, we developed advanced solder materials that are promising replacements for lead based solders, which are banned due to health concerns [7, 8]. All these material systems predominantly fail in a brittle manner with a fracture toughness 1 - 4 MPa m1/2. In these materials, plastic deformation in the vicinity of the artificial notch, is neglected.

In contrast to the aforementioned functional materials, the structural materials of interest are known for their outstanding global fracture toughness. Still, some of the microstructural constituents – either formed while processing (e.g. martensite in dual phase (DP) steels) or introduced during service (e.g. white etching layers) – are known to be brittle. The focus of those studies is to provide reliable fracture properties of these small scale phases e.g. of a single cementite lamellae in pearlitic steel or of white etching layer in rails [9].

Our mechanical experiments are linked to advanced characterization by transmission and scanning electron microscopy, or by synchrotron

[1] Standards proposed by the American Society for Testing and Materials.

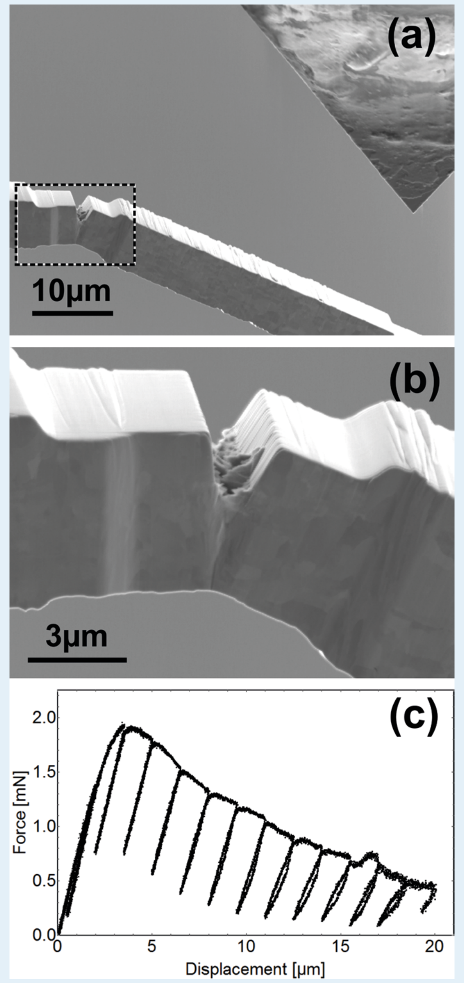

Fig. 1: *Micro fracture experiment on ultra-fine grained tungsten sheets. (a) Scanning electron microscopy image of the experiment with the conductive diamond wedge indenter to the right. (b) Magnified view of the crack and its plastic zone recorded during in situ loading. (c) Force displacement curve showing the decrease in force and unloading stiffness documenting the mechanical impact of crack growth.*

based Laue microdiffraction. Hence, we evaluate the quantitative fracture toughness and determine the toughening mechanisms that are observed by characterization. Furthermore, environmental conditions (elevated temperature, hydrogen loading) are controlled in our experiments. These experiments create new horizons in understanding brittle to ductile transitions [5] or hydrogen embrittlement.

The knowledge of fundamental fracture properties of individual phases and interfaces at the microstructural length scale opens new prospects for the development of damage tolerant structural and functional materials, which is a central focus of the SN department.

One example where the unique data obtained in our studies can directly be used is the project investigated by the collaborative research center TRR 188: "Damage Controlled Forming Processes". Within this research center, we are working on DP steels and investigate the fracture toughness of 1 µm sized martensite islands as well as the fracture toughness of the ferrite-martensite interface. Combined with crystal plasticity modelling, we further identify locations at which damage initiation occurs. On the intermediate time frame, we will use the local toughness values to control and optimize DP microstructures and the strain path during metal forming to avoid damage nucleation. Hence, based on our micromechanical investigations, we can provide devices produced via metal forming with known and optimized damage content. This will significantly reduce the weight and therefore increases sustainability of advanced steel parts produced via metal forming.

Another example which we perform in collaboration with the research group "Materials Science of Mechanical Contacts" is the characterization of the toughness of white etching layers (WELs, see p. 1-2). WELs and similar microstructures cannot be synthesized in bulk form, hence, without the micro fracture experiments developed in the SN department the fracture toughness would not be accessible. Based on these experiments fundamental insights into crack nucleation and propagation in instable microstructures such as the ones formed in the rail-wheel contact can be achieved.

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SN authors: C. Kirchlechner, S. Brinckmann

Awards and Achievements

Dr. Olga Kasian, former postdoctoral researcher in the group "Electrocatalysis", has won the participation in the career development programme "Sign Up! Career building for female post docs." and the leadership of a Helmholtz Young Investigator group.

Dr. Jung Gi Kim, postdoctoral researcher from Korea, received a Humboldt Research Fellowship and joined the group "Alloys for Additive Manufacturing".

Dr. Subin Lee, postdoctoral researcher from Korea, was honoured with a Humboldt Research Fellowship and is now working in the group "Advanced Transmission Electron Microscopy".

Prof. Dierk Raabe, director of the department "Microstructure Physics and Alloy Design", has been honoured by the Royal School of Mines to give the Bauerman Prize Lecture 2019.

Dr. Martin Rabe, postdoctoral researcher in the department "Interface Chemistry and Surface Engineering", won the second best poster award at the "17th International Conference on Organized Molecular Films" in New York, USA.