# Characterization and Properties of Interface-Dominated Materials InMa 2024 Symposium

19-20 November 2024

TZA am Europaplatz
Dennewartstraße 25-27
52068 Aachen

# Contents

# Welcome

Dear Participants,

On behalf of the organizing committee, it is our pleasure to welcome you to the InMa 2024 Symposium on Characterization and Properties of Interface-Dominated Materials, hosted in the dynamic city of Aachen. This event brings together researchers, scientists, and innovators from diverse disciplines, all united by a common pursuit: to deepen our understanding of interfaces and their pivotal role in advancing material properties.

Over the next two days, we invite you to engage in a robust exchange of ideas through keynote presentations, topic-specific sessions, and interactive discussions. Our program includes contributions from leading voices in the field, alongside emerging researchers, fostering a collaborative environment that we hope will spark new insights and partnerships.

We encourage you to explore the thought-provoking topics presented here, from structural and energy materials to advanced methodologies in interface characterization. Each session is an opportunity to broaden perspectives, address current challenges, and push the boundaries of what is possible in interface-dominated materials.

Thank you for being part of this symposium. We look forward to the inspiration and discoveries that will emerge through our shared discussions.

With warm regards,

The InMa 2024 Organizing Committee

# Organizing committee

**Head organizer:** Dr. Nicolas J. Peter
**Co-organizers**: Dr. Jin Wang, Dr. Abril Azocar Guzman, Dr. Bashir Kazimi

# Schedule

| | Tuesday, Nov 19 | | Wednesday, Nov 20 |
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| Until 09:30 | **Arrival** | - | - |
| 09:30 − 10:00 | **Welcome** | 09:00 − 10:00 | **Keynote**: Robert Sinclair |
| 10:00 − 12:00 | **Session 1**: Matteo Ghidelli | 10:00 − 11:40 | **Session 4**: Ralph Spolenak |
| 12:00 − 13:00 | **Lunch** | 11:40 − 12:40 | **Lunch** |
| 13:00 − 15:00 | **Session 2**: Markus Stricker | 12:40 − 14:20 | **Session 5**: Lorenz Romaner |
| 15:00 − 15:30 | **Coffee** | 14:20 − 14:40 | **Coffee** |
| 15:30 − 17:30 | **Session 3**: Jens Bauer | 14:40 − 16:00 | **Session 6**: Siyuan Zhang |
| 17:30 − 18:00 | **Lightning Talks** & **Posters** | 16:00 − 16:20 | **Coffee** |
| 18:00 − 19:00 | **Think-outside-the-box (Incl. beers & snacks)** | 16:20 − 18:00 | **Session 7**: Marc Heggen |
| - | - | 18:00 | **Awards** & **Closing** |

InMa 2024 Symposium Schedule

# Full Program

CT: Contributed Talk, KL: Keynote Lecture, IT: Invited Talk.

## Tuesday, November 19, 2024

| 08:00–9:30 | Arrival | | |
|---|---|---|---|
| 9:30–10:00 | Welcome | | |
| 10:00–10:40 | IT | **Matteo Ghidelli** | Tailoring atomic and microstructural heterogeneities in metallic glass thin films to control and enhance their mechanical properties |
| 10:40–11:00 | CT | **Xi Li** | Strengthening mechanism of Al/Ni multilayers with negative enthalpy of mixing |
| 11:00–11:20 | CT | **Peter Nellessen** | Combinatorial and high throughput characterization of microstructure-property relationships in Ni-Al thin films |
| 11:20–11:40 | CT | **Davide Vacirca** | Tailoring mechanical properties in nanoengineered high entropy alloy thin films |
| 11:40–12:00 | CT | **Arjun Curam** | Defect engineered structural evolution and mechanical characterisation of $Fe_xCoCrNi_{(100-x)}$ complex compositional alloy thin films |
| 12:00–13:00 | Lunch | | |
| 13:00–13:40 | IT | **Markus Stricker** | Data-based approaches for interfaces |

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|---|---|---|---|
| 13:40–14:00 | CT | **Peter Konijnenberg** | Application of ML to the characterization of grain- and phase boundaries by EBSD |
| 14:00–14:20 | CT | **Tom Reclik** | Advancing SEM imaging: integrating deep learning super-resolution for accelerated analysis |
| 14:20–14:40 | CT | **Lukas Berners** | Tailoring damage and plasticity behavior in Mg-4.7Al-2.9Ca with interface governed mechanical properties |
| 14:40–15:00 | CT | **Ude Hangen** | Nanomechanical testing methods to characterize battery materials |
| 15:00–15:30 | **Coffee** | | |
| 15:30–16:10 | IT | **Jens Bauer** | Nanoprinted mechanical metamaterials & materials for nanoprinting |
| 16:10–16:30 | CT | **Wenjuan Cheng** | The role of thin ALD coatings on the performance of micro-/nano-architected meta-materials |
| 16:30–16:50 | CT | **Mohan Kathula** | Effect of defect density and gaseous hydrogen on fatigue behavior of 316L steel manufactured by laser powder bed fusion |
| 16:50–17:10 | CT | **Marco Ezequiel** | Mechanical and electrical properties of amorphous/crystalline nanolaminates for flexible electronic applications |
| 17:10–17:30 | CT | **Guangyi Huang** | Characterization of interfaces in quantum materials using scanning transmission electron microscopy |
| 17:30–18:00 | **Lightning Talks and Posters** | | |
| 18:00 | KL | **Andreas Offenhäusser** | **Think-outside-the-box**: Bioelectronic interfaces – combining biological systems with electronics |

# Wednesday, November 20, 2024

| 9:00–10:00 | KL | Robert Sinclair | Considerations on highly disordered and highly ordered interfaces (with a little twist) |
|---|---|---|---|
| 10:00-10:40 | IT | Ralph Spolenak | Sharp versus diffuse boundaries – the importance of microstructure in reactive multilayers and nanostructures |
| 10:40–11:00 | CT | Gerhard Dehm | Grain boundary phase transitions in fcc metals |
| 11:00–11:20 | CT | Eleonora Isotta | Heat transport at silicon grain boundaries |
| 11:20–11:40 | CT | Hanna Bishara | Local electrical properties of grain boundary complexions in conductive and functional alloys |
| 11:40–12:40 | | Lunch | |
| 12:40–13:00 | CT | Tarik Tandogan | A multi-physics model with dislocation induced nucleation at grain boundaries |
| 13:00–13:40 | IT | Lorenz Romaner | Hybrid modeling of grain boundary chemistry |
| 13:40–14:00 | CT | Sergei Starikov | Disordering complexion transition of grain boundaries: Impact on diffusion and mobility characteristics |
| 14:00–14:20 | CT | Lara Caroline Pereira dos Santos | Understanding interface structure formation in solid-state batteries through ab initio simulations and thermodynamics |
| 14:20–14:40 | | Coffee | |
| 14:40–15:20 | IT | Siyuan Zhang | Operando and correlative techniques to characterize materials degradation |

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|---|---|---|---|
| 15:20–15:40 | CT | **Sri Rathinamani Ramdoss** | Mechanical behavior of a eutectic Mo-Si-Ti alloy |
| 15:40–16:00 | CT | **Kuan Ding** | Microstructural and micromechanical characterization of intergranular oxidation in Fe-15Cr alloy |
| 16:00–16:20 | | **Coffee** | |
| 16:20–17:00 | IT | **Marc Heggen** | In-situ transmission electron microscopy studies of nanoparticle catalysts and complex metallic alloys |
| 17:00–17:20 | CT | **Dylan Jennings** | Mechanistic insights to the atomic-scale coarsening behavior of exsolved catalytic nanoparticles |
| 17:20–17:40 | CT | **Anton Dvydok** | Advanced X-ray nanodiffraction techniques for in-situ and operando tests |
| 17:40–18:00 | CT | **Yug Joshi** | Reversible interfacial Li-oxide formation on germanium and silicon anodes revealed by time-resolved microgravimetry |
| 18:00 | | **Awards & Closing** | |

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## Strengthening mechanism of Al/Ni multilayers with negative enthalpy of mixing

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**Co-authors:** Nicolas J. Peter[2]; Marilaine Moreira de Lima[2]; Ruth Schwaiger[1];

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The interface strengthening effect in nanoscale metallic multilayers could be influenced by the enthalpy of mixing, as determined by the chemical content distribution and the microstructure of interfaces. The Al/Ni multilayers with single layer thickness spanning from 5 nm to 250 nm were fabricated, and their mechanical properties and microstructure were extensively investigated. The Al/Ni multilayers show an ultrahigh peak hardness, 9.5 GPa, which is the highest among the reported fcc/fcc multilayer systems. The microstructure characterization with TEM and STEM-EELS indicates wide interdiffusion at Al/Ni interface and formation of intermetallic bonding at interfaces and grain boundaries in Al/Ni multilayer. A modified confined layer slip (CLP) model was built to introduce the energy change of the trailing dislocation propagating at interface or grain boundary induced by intermetallic, and fits the experimental data very well. Our findings demonstrate that the intermetallic bonding formed at Al/Ni multilayers contributes to a strong interface strengthening effect and compensates the weakening from interface diffusion, which could also be the reason of high peak hardness in other multilayer systems with high negative enthalpy of mixing.

# Combinatorial and high-throughput characterization of microstructure-property relationships in Ni-Al thin films

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Multilayer thin films are an ideal system to study the influence of interfaces on material properties. An interesting multilayer system is Ni-Al because it shows high reactivity and high hardness. In previous work, we were able to identify an intermixing zone at the Ni-Al interfaces, which we refer to as 3D-interface, and were able to identify a change in bonding at these interfaces towards more "intermetallic-like" bonding. However, it remains unclear if the intermixing alone would be sufficient to explain the high hardness of this multilayer system or if the change in bonding at the interface is required in addition. To shed light on the origin of the high hardness values, in the present study we study sputtered Ni-Al thin films in a combinatorial and high-throughput approach over a large compositional space.

A wafer with 47 different Ni-Al concentrations along a gradient was produced by magnetron co-sputtering and analyzed by energy-dispersive X-ray spectroscopy (EDS), X-ray diffraction (XRD), nanoindentation, transmission electron microscopy (TEM) and 4D scanning transmission electron microscopy (4D-STEM). Ni contents ranging from 44.4 to 72.9 at.% were identified across the concentration gradient, which revealed significant microstructural changes from equiaxed grains to columnar grains. Grain sizes varied between few tens of nanometers in the Ni-rich samples, while anisotropic grains with diameters of several hundred nanometers in growth direction were found in the rest of the samples. Complementing the overall phase composition of each sample from XRD, 4D-STEM provided more spatially resolved information on the phase distribution and content as a function of concentration. Overall, this study presents the formation of a B2-NiAl and A1-Ni(Al) solid solution phase mixture, irrespective of the Ni and Al concentrations. The ratio of the two phases is concentration-dependent, while hardness remains high and almost unaffected by composition. We introduce a data-informed model, which is able to describe the hardness of the Ni-Al thin films across the studied concentration range. Our work indicates that intermixing zones can result in high hardness of the multilayer stack not only due to solid solution strengthening, but depending on the concentration, changes in bonding may provide an additional strengthening contribution.

# Tailoring mechanical properties in nanoengineered high entropy alloy thin films

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High entropy alloy thin films (HEA-TFs) have gained attention due to their interesting combination of properties such as high hardness and thermal stability while offering a wide range of chemical compositions, leading to potential applications for microscale electrical devices or high-performance coatings [1]. However, a nanoengineering approach based on the synthesis of new film architectures with controllable nanoscale features (i.e., grain size, nanolaminate structures) to improve mechanical properties is still an open challenge, requiring the implementation of new synthesis routes.

Here, nanocrystalline CoCrCuFeNi HEA-TFs with a wide range of microstructures have been deposited using pulsed laser deposition (PLD). By controlling the background pressure, a transition from compact to nanogranular morphology was observed for deposition pressures > 1 Pa, leading to a decrease in density and grain size ($6.81\,g/cm^3$ and 11 nm, respectively) compared to the compact counterpart ($7.72\,g/cm^3$ and 40 nm, respectively). Both compact and nanogranular films by PLD show increased hardness ($\approx 11$ GPa) compared to sputter-deposited films ($\approx 8.3$ GPa) due to their smaller grain size. Post-thermal annealing treatments reveal grain coarsening (up to 60 nm at 460°C) and segregation of a secondary BCC phase starting at 400°C for both compact and nanogranular films. However, nanogranular films maintain a lower grain size during annealing (< 20 nm), thus retaining higher hardness of 8.5 GPa (Hall-Petch strengthening).

Then, we will focus on Al/HEA nanolaminates by PLD and magnetron sputtering with bilayer period ($\Lambda$) ranging from 200 nm down to 2.5 nm, aiming to block the propagation of dislocations by controlling the interface density to improve hardness and yield strength [2]. Nanolaminates with different atomic compositions have been fabricated involving semi-coherent (FCC/FCC Al/CoCrCuFeNi) and incoherent (FCC/BCC Al/Al$_5$(CoCrCuFeNi)$_{75}$) interfaces to further hinder cross-layer dislocation propagation. Among the main results, in situ micropillar compression tests show that incoherent interfaces have greater capability to improve yield strength (up to 4.5 GPa), while showing great plastic deformability with no appearance of cracks even at 30% deformation.

[1] N. I. M. Nadzri *et al.*, Coatings, 12, 2022
[2] A. Sáenz-Trevizo *et al.*, 31, 2020

# Mechanical and Electrical Properties of Amorphous/Crystalline Nanolaminates for Flexible Electronic Applications

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**Co-authors:** Evgeniy Boltynjuk[2]; Julia Ivanisenko[2]; Damien Faurie[1]; Philippe Djemia[1]; Horst Hahn[2]; Matteo Ghidelli[1]

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The development of flexible electronics is driving the search for new materials that offer a combination of exceptional electrical and mechanical properties [1]. Thin film metallic glasses (TFMGs) are attractive due to their superior mechanical properties, low and negative temperature coefficient of electrical resistivity, stability of electrical properties across thickness variations, and strong fatigue resistance. However, their use in flexible electronics is limited by their higher electrical resistivity compared to crystalline materials and their macroscopic brittleness, driven by shear bands (SBs) formation [2]. Nanostructuring strategies, such as nanolaminating can hinder these downsides. Nevertheless, unresolved scientific issues involving understanding the deformation mechanisms for nanostructured crystal/glass and developing effective synthesis strategies to improve the mechanical and electrical properties simultaneously are still present.

This study presents amorphous/crystalline ZrCu/Fe-BCC nanolaminates, which offer enhanced mechanical and electrical properties through controlled ZrCu fractions (0.5 to 0.9) and bilayer periods (10 to 40 nm). We demonstrate that the number of ZrCu/Fe interfaces significantly affects hardness, with the nanolaminates achieving high hardness values up to 9.6 GPa, substantially higher than the individual components. Additionally, these nanolaminates show electrical resistivity as low as 36 µOhm.cm, similar to that of pure Fe.

In addition, we studied the deformation mechanism using micropillar compression tests. These revealed that the 20 nm Fe/20 nm ZrCu nanolaminate exhibits high strain without failure, unlike pure ZrCu, which shows shear band propagation at low strain. The superior mechanical behavior of the nanolaminates is attributed to the inhibition of the shear band propagation by their interfaces.

Furthermore, we conducted tensile tests on samples deposited on a flexible polymeric substrate to assess the potential of these materials in flexible electronics applications. The nanolaminates demonstrated crack onset strains of up to 1.36% and promising high crack propagation strains of up to 13%.

Overall, our findings underline the critical role of nanostructuring in improving both the mechanical and electrical properties of nanolaminates, expanding their potential for use in flexible electronics.

[1] Corzo, D. et. al., Frontiers in Electronics 1, (2020).
[2] Lee, S. et al., Nano Lett 20, 4872–4881 (2020).

# Defect engineered structural evolution and mechanical characterisation of $Fe_xCoCrNi_{(100-x)}$ complex compositional alloy thin films

**Author:** Arjun Curam[1]

**Co-authors:** Marco Ezequiel[2]; Vianney Mille[1]; Gregory Abadias[3]; Philippe Djemia[1]; Christian Ricolleau[4]; Matteo Ghidelli[1]

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Complex compositional alloy thin films (CCA-TFs) are gaining interest for their large strength/ductility balance, owing to FCC nanocrystalline structure [1]. CoCrNi reported hardness (H) and elastic modulus (E) up to 9 and 180 GPa, respectively, due to a columnar structure with planar defects [2]. Adding Fe stabilized the FCC phase and a small grain size of 20 nm, mildly increasing H up to 10 GPa [3]. However, research on CCA-TFs is still in its early stages, particularly regarding their growth mechanisms and relationship with mechanical behavior, while a nanoengineered approach to develop new thin film nanoarchitectures with boosted mechanical properties remains an open field.

Here, we synthesized CoCrNi and $Fe_xCoCrNi_{(100-x)}$ CCA-TFs by magnetron sputtering, varying their thickness and Fe content. CoCrNi shows a columnar structure with an average grain size of 26 nm and (111) textured FCC structure and minor HCP phase. Nanoindentation and optoacoustic techniques report H = 9.7 GPa and E = 204.4 GPa, in line with the literature [3].

Moreover, adding Fe retained the columnar structure while stabilizing the FCC phase, reporting a preferred (111) orientation without phase segregation. H and E decrease down to 7.6 and 185 GPa due to fewer defects like stacking faults (SFs) and twins, owing to a higher stacking fault energy of 32.5 mJ m$^{-2}$ [4].

Finally, we synthesized 1.3 µm thick CoCrNi/Fe (FCC/BCC) nanolaminates with bi-layer periods (Λ) of 35 and 70 nm, featuring incoherent interfaces aimed at blocking dislocations and crack propagation. We show high adhesion at the interfaces with limited crack formation during indentation, while compression of micropillars resulted in high yield strengths (5.4 GPa) without fracture up to 12% and high plasticity up to 40% due to the high density of defects, increased interfaces, and thickness confinement. Overall, our study sheds light on the structure–mechanical properties of CoCrNi, $Fe_xCoCrNi_{(100-x)}$ films, and CoCrNi/Fe nanolaminates, with potential applications in hard coatings and microelectronics.

[1] Yuanying Y, et al., J. Mater. Res. Technol, 21, 2022

[2] Yvonne (Yi-Ting) Lin, et al., Surf. Coat. Technol., Volume 424, 2021

[3] Nagy, P et al., Surf. Coat. Technol., Volume 386, 2020

[4] Y. Wang et al., Acta Mater, 154, 2018

# Application of ML to the Characterization of Grain- and Phase Boundaries by EBSD

**Author:** Peter Konijnenberg[1]

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Electron BackScatter Diffraction (EBSD) is a rapidly evolving, ubiquitous technique for analytical scanning electron microscopy. On the software side, a key element of this technique consists of an algorithm to process a stream of electron backscatter diffraction images (Kikuchi patterns) in a high throughput setup. Traditionally, this comprises a step of detecting Kikuchi bands by a Hough-transform followed by a crystallographic identification step (indexing), e.g., by a voting approach. Over the last decade, significant advancements were reported via the use of pattern matching algorithms that essentially use the full information content by cross-correlating the acquired patterns with a forward-simulation, see e.g. [1,2]. Apart from some early attempts [3], the application of neural networks to the entire pattern analysis process was reported only relatively recently, see e.g. [4,5], where forward-simulated patterns are utilized to train a convolutional neural network (CNN), which then can be used for the task of analyzing acquired patterns.

In this contribution, we report on the development and application of such an ML-based approach to the analysis of Kikuchi patterns specifically at grain and phase boundaries.

In the vicinity of grain boundaries, depending on the grain boundary inclination w.r.t. the electron beam, a superposition of two Kikuchi patterns from adjacent grains is often observed. In the case of Hough-based analysis, overlapping Kikuchi patterns frequently result in a zone of mis- or non-indexed map points along grain boundaries. We demonstrate and evaluate how the ML-method deals with these overlapping patterns.

In polar materials, anti-phase boundaries (APBs) separate domains of opposing chemical ordering. Due to Friedel's rule, classical EBSD techniques, which are based on the kinematical pattern model, are bound to fail on these boundaries. Hence, only the orientation of the overarching Laue group can be determined. As shown by Winkelmann et al. [6], a point-group correct orientation analysis can overcome this through the additional use of pattern matching with dynamically simulated patterns. We demonstrate that the ML-based method is able to achieve this in a single analysis step.

[1] Chen, Y. H., et al. Microscopy and Microanalysis 21.3 (2015): 739-752.
[2] Hielscher, R. et al. Ultramicroscopy 207 (2019): 112836.
[3] Schwarzer, R.A., and Sukkau, J. Advanced Engineering Materials 5.8 (2003): 601-606.
[4] Dipendra, J. et al. Microscopy and Microanalysis 24.5 (2018): 497-502.
[5] Ding, Z. et al. Acta Materialia 199 (2020): 370-382
[6] Winkelmann, A. and Nolze, G. Applied Physics Letters 106.7 (2015).

# Advancing SEM Imaging: Integrating Deep Learning Super-Resolution for Accelerated Analysis

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Performing high-resolution large-area scanning electron microscopy for the analysis of micro- and nanoscale features poses significant challenges, including prolonged scanning durations and potential sample degradation due to extended electron beam exposure. This is further amplified when the sample needs to be imaged multiple times in different states, as in in-situ experiments. Here, we develop a novel method based on the texture transformer network for super-resolution to enhance the resolution of electron micrographs using artificial intelligence, thereby accelerating the image acquisition process by a factor of up to 16. We evaluate the performance of this approach on two types of steel: a dual-phase steel and a case-hardening steel. Using our approach can significantly speed up the imaging process, enabling discoveries of new physics with high statistical relevance.

# Tailoring damage and plasticity behaviour in Mg-4.7Al-2.9Ca with interface governed mechanical properties

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**Co-authors:** Setareh Medghalchi[1]; Pei-Ling Sun[1]; Sandra Korte-Kerzel[1];

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The Mg-Al-Ca system has provided promising candidates for creep-resistant Mg alloys due to the formation of a connected network of intermetallic Laves phases. However, this network is inherently brittle and primarily accommodates plastic deformation by cracking at strain rates of $5 \times 10^{-4}$ [1]. At lower strain rates of $5 \times 10^{-6}$, decohesion of the Mg matrix from the Laves phase occurs [2]. Additionally, deformation based on dislocations is observed in the Laves phase, but only as a minority phenomenon [3].

In the present study, we show that the dominant failure mechanism, cracking of the Laves phase, can be reduced by refining the microstructure using the cooling rate as a design parameter. We utilize the neural network YoloV5, trained to identify Laves phase cracks, decohesion sites, and artifacts on large-scale panoramic SEM images. Additionally, we identified slip traces in the Laves phase by manually labeling the panoramic SEM images. The results show that the cracking mechanism is predominant at lower cooling rates and, therefore, in coarser microstructures. In contrast, at higher cooling rates, the microstructure is refined and exhibits more slip transmission compared to cracking.

[1] M. Zubair et al., Mater. Sci. Eng. A 758 (2019), 272-283
[2] S. Medghalchi et al., Adv. Eng. Mater. 25 (2023), 2300956
[3] M. Zubair et al., Mater. Des. 210 (2021), 110113

# Nanomechanical Testing Methods to Characterize Battery Materials

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Battery performance degradation over time is contingent on chemical and mechanical changes at material interfaces. This degradation can be better understood through nanomechanical (nanoindentation) testing, which must be performed in high-purity environments due to the reactive nature of battery materials. Batteries, ubiquitous in modern life, degrade in performance with each charge-discharge cycle, often due to changes at the interfaces. Understanding and controlling these changes are essential to increasing energy density, the number of charge-discharge cycles, and safety while decreasing cost and weight.

This study focuses on nanomechanical (nanoindentation) materials testing in low oxygen, low moisture environments. It presents measurements of hardness and modulus on current-generation lithium-ion cathodes, with a primary focus on solid electrolyte battery components. These include compressed sulfide powder-based solid electrolytes undergoing densification and intergranular fracture, soft lithium and sodium foils with a strong rate effect, and density variations in half-cell solid-state lithium batteries. The techniques discussed will enable researchers to quantify highly localized mechanical degradation, facilitating the development and integration of new materials for next-generation batteries.

# The Role of Thin ALD Coatings on the Performance of Micro-/Nano-architected Meta-materials

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Two-photon polymerization-direct Laser Writing (TPP-DLW) derived ceramic metamaterials face challenges in humid conditions, showing a 20% reduction in fracture toughness at high relative humidity (RH > 60%) due to surface defects from fabrication. This research explores improving the environmental reliability of these materials through Atomic Layer Deposition (ALD) coatings on ceramic micro-pillars. A uniform 50 nm layer of $Al_2O_3$ was applied using Plasma Enhanced-ALD (200 °C) and Thermal ALD (200 °C and 350 °C). The effects of the coating on fracture toughness and crack propagation in glassy carbon (GC) were evaluated under various relative humidity levels (below 5% and above 60%) through pillar splitting tests and precise stress measurements using Focused Ion Beam (FIB) ring-core analysis. A parallel investigation on lithographically produced fused quartz micro-pillars treated with the same ALD parameters served as a comparison to assess the coatings' effectiveness in enhancing fracture toughness. Cohesive element simulations provided insights into the role of ALD-induced residual stress in both GC and silica systems, supporting the experimental findings.

The results show that the 50 nm $Al_2O_3$ coating effectively mitigated the negative effects of humidity on GC pillars, restoring fracture toughness to levels comparable to dry conditions, with an improvement of about 28%. Residual stresses induced by ALD, varying by deposition temperature, played a critical role in crack initiation and propagation. In particular, the reduction in fracture toughness in coated pillars, compared to undefective, uncoated pillars under similar humid conditions, was attributed to premature crack tip opening caused by surface defects.

The findings further demonstrated how ALD-induced stresses significantly enhanced the fracture toughness of silica structures, compared to the baseline crack resistance (0.643 MPa$\sqrt{m}$). A 50 nm coating at 200 °C increased toughness by 134%, while a 100 nm coating at 300 °C achieved a 165% improvement. This illustrates how interface engineering (deposition temperature and induced stresses from ALD coatings) can fine-tune fracture toughness in 3D TPP micro-ceramics, depending highly on the substrate material and surface defects (likely missing in lithography silica structures).

# Effect of defect density and gaseous hydrogen on fatigue behavior of 316L steel manufactured by laser powder bed fusion

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The integration of hydrogen as a clean energy carrier offers a promising approach to decarbonize many sectors in the pursuit of global carbon neutrality. Metallic components exposed to hydrogen environments during service can experience significant degradation in mechanical properties. Recently, metal additive manufacturing, especially Laser Powder Bed Fusion (LPBF), has been employed for producing numerous commercial metallic alloys, highlighting the importance of understanding the relationship between process parameters, defect density, and resulting microstructure-property behavior under hydrogen influence.

In this study, hollow fatigue specimens of 316L stainless steel were fabricated using LPBF with two different volumetric energy densities to control the defect density. The hollow specimens were then filled with hydrogen gas up to 20 bar and subjected to low-cycle fatigue testing to evaluate the degradation in fatigue life and investigate the deformation mechanisms induced by hydrogen exposure. Advanced characterization techniques, including scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX), electron backscatter diffraction (EBSD), and transmission electron microscopy (TEM), were employed to analyze the microstructural changes and fatigue deformation mechanisms. Fracture surfaces were examined to reveal the characteristics of fatigue fracture under hydrogen influence.

In general, specimens manufactured with higher volumetric energy density showed longer fatigue lifetimes at all tested stress levels, attributed to reduced defects, particularly of the lack-of-fusion type. Additionally, the inner surfaces exposed to gaseous hydrogen exhibited changes in deformation mechanisms, including deformation twins and austenite-to-martensite transformation, compared to outer surfaces. These hydrogen-induced changes in deformation mechanisms are crucial in determining fatigue lifetime, particularly at high stress levels.

The findings provide insights into the role of defect density and deformation mechanisms in determining the fatigue lifetime of LPBF-manufactured 316L stainless steel under hydrogen exposure, contributing to improved process optimization and material performance in hydrogen environments.

# Characterization of interfaces in quantum materials using scanning transmission electron microscopy

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In recent years, interface engineering has emerged as one of the most active areas in developing novel quantum materials. For example, two-dimensional van der Waals materials can break strong bonds between neighboring layers, enabling epitaxial growth of various films on previously incompatible substrates despite significant lattice mismatches. Furthermore, precise atomic arrangement at interfaces can alter local electronic and spin structures, generating unconventional quantum properties [1-4].

In this presentation, we will introduce the study of interfaces in quantum materials using aberration-corrected scanning transmission electron microscopy. We will explore the structure, composition, and electron charge transfers in heterointerface superconductivity systems. We'll also demonstrate how 2D van der Waals materials can be used to grow unconventional heterostructures for studying quantum phenomena. Additionally, we'll show rapid aspect ratio measurement of one-dimensional quantum structures using quantitative scanning transmission electron microscopy. Finally, we will report on the characterization of ultra-thin ice film growth in a high-vacuum environment.

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# Grain boundary phase transitions in fcc metals

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The thermodynamic concept of phase transformations of bulk materials is well established and forms the basis for material and microstructure design through phase diagrams. However, it is largely unknown in the materials community that grain boundaries (and interfaces) can also undergo phase transitions—recently termed "complexions" to avoid confusion with bulk phase transitions [1]. The thermodynamic concepts for complexions have been developed over the last decades, though direct experimental evidence is rare due to the difficulty of accessing the atomic structures of grain boundaries.

Highly textured metallic thin films provide an opportunity to study structure and phase transitions as a function of temperature and chemical composition. Scanning transmission electron microscopy studies show that even in pure copper, grain boundary phases can coexist [2-4], although atomistic simulations of the free grain boundary energy indicate that coexistence at ambient temperature is metastable, suggesting a sluggish transformation. Additionally, different grain boundary phases exhibit distinct segregation behaviors upon alloying (see, e.g., [5]).

While there has been substantial indirect evidence that grain boundary phases and their transitions influence material properties, only limited direct correlations have been established. This presentation reviews the current understanding of grain boundary phases and their impact on properties, with a focus on aluminum and copper-based metals.

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# Heat transport at silicon grain boundaries

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Engineering microstructural defects, like grain boundaries, offers superior control over transport properties in energy materials. However, technological advancement requires establishing microstructure-property relations at micron or finer scales, where most of these defects operate. Here, we provide the first experimental evidence of thermal resistance for individual silicon grain boundaries, estimated with a Gibbs excess approach. Coincident site lattice boundaries exhibit uniform excess thermal resistance along the same boundary but notable variations from one boundary to another. Boundaries associated with low interface energy generally exhibit lower resistances, aligning with theoretical expectations and previous simulations, but several exceptions were observed.

Transmission electron microscopy reveals that factors like interface roughness and the presence of nanotwinning can significantly alter the observed resistance, which ranges from 0 to 2.3 $m^2K/GW$ (noise floor 0.15 $m^2K/GW$). In stark contrast, significantly larger and less uniform values—on the order of 5-30 $m^2K/GW$—are found for high-angle boundaries in spark-plasma-sintered polycrystalline silicon. Further, finite element analysis suggests that boundary planes that strongly deviate from the sample vertical (more than 45°) can show up to 3 times larger excess resistance. Direct correlations of properties with individual defects enable the design of materials with superior thermal performance for applications in energy harvesting and heat management.

[1] E. Isotta, S. Jiang, R. Bueno-Villoro, R. Nagahiro, K. Maeda, D. A. Mattlat, A. R. Odufisan, A. Zevalkink, J. Shiomi, S. Zhang, C. Scheu, G. J. Snyder, O. Balogun, Adv Funct Mater 2024, DOI 10.1002/adfm.202405413.

# Local electrical properties of grain boundary complexions in conductive and functional alloys

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Grain boundaries (GBs) significantly affect the electrical properties of metallic and semiconductor materials. The interfacial resistivity of GBs is generally considered an accumulative property across all GBs within a material, averaging over multiple GB types. However, different boundary types exhibit distinct structural and chemical characteristics, leading to a spectrum of electrical properties. Despite this, the relationship between a boundary's characteristics and its electron transport properties is not well understood. Uncovering the contributions of different GB types to electrical resistivity could enable novel defect engineering approaches to optimize the performance of conductors and functional alloys. This talk presents studies on the impact of GB structure on its resistivity, with a focus on the local electrical characterization of individual GB segments.

The presentation introduces an experimental procedure for measuring the local electrical resistivities of GB segments with high sensitivity and spatial resolution in-situ scanning electron microscopy (SEM). Simultaneously, these segments are characterized by electron backscatter diffraction (EBSD), transmission electron microscopy (TEM), energy dispersive spectroscopy (EDS), and atom probe tomography (APT), as well as molecular dynamics (MD) simulations. The measured electrical properties are linked with the structural characteristics and thermodynamic excess properties of the boundaries. Multiple material systems will be discussed, including pure Cu, dilute Cu alloys, aluminum alloys, and Heusler thermoelectric alloys. In addition to the resistive nature of GBs, the lecture reports unexpected trends of conductive complexions in GBs and stacking faults in Heusler alloys, attributed to segregation phenomena that make these complexions more conductive than the grain interior.

This talk provides insights into the methodology for assessing local electrical resistivity of well-defined complexions. It demonstrates the influence of structural and thermodynamic properties of internal interfaces on the electrical properties of metallic and thermoelectric materials. The novel results contribute to a better understanding of defects' resistivity and open new horizons for knowledge-based defect engineering in smart materials. This research shows promise for applications in phase boundaries and internal interfaces.

# Disordering complexion transition of grain boundaries: Impact on diffusion and mobility characteristics

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Many microstructural changes in structural materials are influenced by the kinetic behavior of grain boundaries (GBs) and interfaces. Consequently, accurately describing the kinetic characteristics of these defects is crucial for developing physically-informed models of material evolution. Classical atomistic simulations provide a valuable approach to uncover key details of defect properties. This work focuses on temperature-induced order-order and order-disorder transitions of GBs and their impact on diffusion and mobility characteristics.

The study examined several metals (Ni, Fe, Nb, Mo, and W) using new reliable interatomic potentials developed with a machine-learning-based force-matching technique. Simulations revealed that atomic self-diffusion along tilt GBs at low temperatures is primarily driven by self-interstitial atoms. However, as the temperature increases, the GB diffusion mechanism shifts to a more complex exchange process similar to diffusion in a liquid, unassociated with a specific defect. This transition is linked to the disordering complexion transition of GBs, which also significantly affects GB mobility.

# A Multi-Physics Model With Dislocation Induced Nucleation At Grain Boundaries

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The granular microstructure of metals evolves extensively during thermomechanical treatment through viscoplastic deformation and recrystallization. Grain boundaries can migrate due to increased mobility, while severe plastic deformation can lead to the formation of subgrains and localized deformation bands. During heat treatment following plastic deformation, new dislocation-free grains nucleate and grow into neighboring grains, reducing the total energy. Defects such as grain boundaries and second-phase particles serve as prime nucleation sites due to strong lattice orientation gradients and high dislocation concentrations.

Numerical modeling of this coupled evolution problem is typically handled through separate frameworks for mechanical deformation and grain boundary migration, using a staggered approach. Nucleation is often incorporated as an intermediate step, with nuclei inserted at possible sites based on criteria like critical dislocation density, stress, or strain. In this work, we present a unified and thermodynamically consistent field theory where grains can nucleate spontaneously at pre-existing grain boundaries as a result of plastic deformation. The model couples Cosserat crystal plasticity with a Henry-Mellenthin-Plapp-type orientation phase field, capturing both curvature- and dislocation-driven grain boundary migration.

The model's capabilities are demonstrated through numerical examples with periodic bicrystals and polycrystals, where strain-induced boundary migration, sub-grain growth, and coalescence mechanisms are observed.

# Understanding interface structure formation in solid-state batteries through ab initio simulations and thermodynamics

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Solid-state batteries are promising candidates for the next generation of electrochemical cells. However, the poor interface between the lithium metal anode (LMA) and the solid electrolyte (SE) is a major challenge limiting their broader adoption. In a simple, yet innovative approach, we mix solid-state lithium (Li) and sodium (Na) metals to create a meta-stable, homogeneous LiNa anode for use with $Li_{6.45}Al_{0.05}La_3Zr_{1.6}Ta_{0.4}O_{12}$ (LLZO) SEs. During Li stripping, the LiNa anode forms a self-organized 3D microstructure that significantly improves the LMA/LLZO interface.

We propose that this 3D microstructure is generated through spinodal decomposition during cycling, as the Na content gradually increases. This phenomenon is the focus of our current study. Using ab initio calculations, we explore the thermodynamics of the Li-Na system, extracting system energetics and predicting the low-temperature phase diagram, where Li and Na are practically immiscible. The onset of spontaneous phase separation is determined both with and without considering elastic mechanical effects, providing a theoretical composition range where such interface structures are expected to form.

# Mechanical behavior of a eutectic Mo-Si-Ti alloy

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The demand for new high-temperature materials to increase operating temperatures and thereby enhance the efficiency of energy conversion systems is growing [1]. While Ni-based superalloys offer an excellent combination of high strength [2,3] and high-temperature oxidation resistance [4], their application is physically limited by a solidus temperature of approximately 1300°C [2]. Mo-Si-Ti alloys have garnered attention for their remarkable oxidation and creep resistance [5,6], making them suitable for applications at temperatures beyond those of Ni-based alloys. Specifically, the eutectic composition Mo-20Si-52.8Ti (at%) provides a well-balanced combination of properties, offering both effective oxidation resistance and improved creep resistance. This composition yields a body-centered cubic (BCC) disordered Mo-rich solid solution and an intermetallic hexagonal $(Ti,Mo)_5Si_3$ phase. However, due to their low ductility at room temperature with a brittle-to-ductile transition temperature (BDTT) around 1100 °C [7], their applicability is currently limited.

Three distinct microstructures of eutectic Mo-Si-Ti were obtained from different processing routes. The mechanical performance of these microstructures is discussed based on macro-mechanical and micromechanical tests.

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# Microstructural and micromechanical characterization of intergranular oxidation in Fe-15Cr alloy

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Fe-Cr alloys show great potential as interconnect materials for solid oxide fuel cells (SOFCs) at temperatures up to 800 °C, with Cr providing excellent oxidation resistance through the formation of a protective chromium oxide layer. However, internal brittle oxides that form at grain boundaries (GBs) can lead to severe cracking and material failure. This study reports the formation of internal GB oxide in an Fe-15wt.%Cr alloy, with a detailed microstructural and chemical characterization of the GBs before and after oxidation.

Before oxidation, the grain structure and intergranular carbides were examined. Post-oxidation, internal GB oxide (IGBO) with pores was observed along the GBs, characterized in detail using electron microscopy, revealing GB oxide formation with significant penetration depth. Elemental mapping indicated that the IGBO was chromium-rich. Additionally, potential mechanisms leading to intergranular oxidation were discussed.

Furthermore, both ex-situ and in-situ wedge-indentation tests were conducted to investigate the mechanical behavior of IGBO. Together with Finite Element Method (FEM) simulation, results demonstrated that IGBO is prone to cracking under mechanical loading, which negatively impacts mechanical reliability. Our findings suggest that processing the alloy with higher purity raw materials and better homogenization may help minimize the detrimental effects of intergranular oxidation in Fe-Cr-based interconnects for SOFCs.

# Mechanistic Insights to the Atomic-Scale Coarsening Behavior of Exsolved Catalytic Nanoparticles

**Authors:** Dylan Jennings[1]; Moritz L. Weber[1]; Ansgar Meise[1]; Tobias Binninger[1]; Moritz Kindelmann[1]; Marc Heggen[1]; Joachim Mayer[1]; Wolfgang Rheinheimer[2]

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The development of nanostructured composite materials is critical for improving the efficiency of various heterogeneous catalytic processes. Metal exsolution offers a promising synthesis route for forming oxide-supported metal catalysts, enabling the creation of well-dispersed and highly active metal nanoparticles. Although exsolution is often suggested as a method for enhancing particle stability, thereby leading to catalytic systems with increased efficiency and extended lifetimes, recent work has indicated limitations to the thermal stability of exsolved nanoparticles. Therefore, a fundamental understanding of the factors impacting the stability of exsolved nanoparticles is essential for optimizing catalytic systems.

In this work, environmental scanning transmission electron microscopy (STEM) is combined with a careful sample preparation process to achieve atomic-resolution secondary electron (SE) imaging during in situ STEM exsolution experiments. An epitaxially grown Ni-doped strontium titanate thin film, with a nanostructure comprising a Ni-doped strontium titanate matrix and embedded heteroepitaxial NiO nanocolumns, is utilized for in situ experiments. Initial characterization of exsolution from this material reveals the formation of two distinct populations of Ni nanoparticles—those precipitating above second-phase nanocolumns and those nucleating above the matrix.

The dynamics of exsolved nanoparticles post-exsolution are observed by in situ STEM, with a focus on the behaviors of the two particle types in relation to Ostwald Ripening and particle migration. Key insights into particle migration include measurements of random-walk kinetics and particle shape changes during movement, which challenge classical models of the atomistic mechanism of particle migration. Additionally, the Ostwald Ripening-correlated particle dissolution process is captured in situ with atomic-resolution SE imaging, revealing surface modifications immediately following particle dissolution.

# Reversible interfacial Li-oxide formation on germanium and silicon anodes revealed by time-resolved microgravimetry

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Germanium has recently gained attention as a promising candidate for next-generation Li-ion anodes due to its high capacity, superior conductivity, and faster lithium-ion diffusivity compared to silicon. However, uncertainties surrounding the Li-storage mechanism and the formation of a solid electrolyte interface (SEI) have hindered its further development. In this study, operando microgravimetry was performed on Ge electrodes to quantify the SEI mass (irreversibly deposited) and to distinguish it from reversibly cycled species, assessing their individual contributions to total capacity.

Time-resolved mass spectra, obtained by correlating gravimetry with charge flux, revealed that $Li_2O$, along with Li, acts as a reversibly cycled species, significantly contributing to the electrochemical capacity. The amount of cycled $Li_2O$ depends on both the cycling rate and the thickness of the anode, with a decrease in $Li_2O$ observed if pronounced anode cracking occurs. A direct comparison between Si and Ge indicates that the amount of reversible $Li_2O$ is primarily governed by lithium diffusivity into the electrodes. This study provides a general mechanism for balancing interfacial and bulk Li storage, either as an oxide or through alloying, respectively.

# Advanced X-ray nanodiffraction techniques for in-situ and operando tests

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Novel materials and their properties at the nanometer scale present broad prospects for future applications in catalysis, sensor technology, energy conservation, sustainability, and high-speed electronics. Detailed investigations, including in-situ and operando tests, are essential for realizing these potential applications. Integrating the sample environment with experimental techniques involves addressing various factors, from space requirements for in-situ setups to specific material characteristics. X-ray nanodiffraction provides a powerful, non-destructive method suitable for analyzing a wide range of materials.

Modern X-ray optics enable a small X-ray beam size with a substantial working distance, offering ideal conditions for integration with additional setups. The Nanofocus Endstation at the P03 beamline of the PETRA III synchrotron (DESY, Hamburg) routinely delivers a 250x250 $nm^2$ synchrotron beam with a 9 cm working distance, compatible with various sample environments for mechanical, thermal, and electrical testing. This presentation will cover the technical specifications, capabilities, and in-situ facilities of the instrument, illustrated by results from exemplary experiments.

# List of Abstracts – Posters

## Electro-chemo-mechanical behavior of a layered cathode material upon cycling

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The mechanical characteristics of lithium-ion cathode materials are crucial for determining battery performance metrics such as durability, cycle life, and safety, particularly under external pressure, as in all-solid-state batteries. This study examines $LiCoO_2$ (LCO), a commonly used hexagonal layer-structured cathode material for lithium-ion batteries, focusing on its mechanical properties during de-/lithiation using sputter-deposited thin films and nanoindentation.

The experimental Young's modulus in the fully lithiated state is quantified to be 337.1 ± 8.7 GPa and 267.9 ± 7.2 GPa for the (101) and (003) lattice orientations, respectively. A significant texture-dependent decrease in Young's modulus upon lithium deintercalation is observed, likely due to modifications in bonding interactions between cobalt oxide layers. Reduced Li content also increases the relative contribution of plastic deformation, suggesting that dislocation glide becomes easier in deintercalated states. With extensive cycling, the Young's modulus in higher lithiated charge-states decreases considerably, likely due to irreversible phase transitions. Conversely, a notable increase in plastic hardness is observed with cycling, understood as work hardening.

This study provides valuable insights into the dynamic changes in mechanical properties during electrochemical cycling of $LiCoO_2$, paving the way for understanding similar behavior in other layered cathode materials.

# Investigation on Photocorrosion of TiO$_2$ during the Photoelectrochemical Process by In-Situ ICP-MS Characterization

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With the growing environmental concerns and increasing energy demand, renewable and clean energy sources have become critical research areas. Hydrogen, an abundant and high-energy-density fuel, is particularly desirable due to its clean byproduct—water. In 1972, Fujishima and Honda discovered that hydrogen could be generated from water splitting on a TiO$_2$ photoelectrode under UV light. However, TiO$_2$ has a low energy conversion efficiency for photo(electro)catalytic processes due to fast electron–hole recombination and its large bandgap (3-3.2 eV). Various strategies, including reduction treatments that induce oxygen vacancies, have been developed to improve light absorption, charge separation, and photocatalytic performance in TiO$_2$.

Though TiO$_2$ is generally considered stable due to its redox potential, which is favorable for driving desired redox reactions without degrading the material, actual photocorrosion behavior depends on kinetic factors such as illumination intensity, electrolyte composition, radiation absorption, and mass/charge transport. In this study, we used inductively coupled plasma mass spectrometry (ICP-MS) to detect dissolution products of TiO$_2$ photoelectrodes during photoelectrochemical (PEC) water splitting. Results reveal that the TiO$_2$ photoanode undergoes corrosion from oxidation by photogenerated holes, with photodissolution influenced by the applied potential.

Our findings provide insights into how the atomic-scale structure of TiO$_2$ correlates with its PEC performance and photocorrosion behavior, offering a deeper understanding of the structure-activity-stability relationship in photoelectrocatalysis. ICP-MS analysis under solar water splitting conditions exposes chemical instabilities not predicted by thermodynamic stability considerations, as represented by the Pourbaix diagram. This study experimentally demonstrates the competition between water splitting and semiconductor oxidation in the photochemical corrosion pathway of TiO$_2$ thin films.

# Three-dimensional Reconstruction for Microstructure and Micromechanical Property by Nanoindentation and Machine Learning Prediction

**Authors:** Ruomeng Chen[1]; Steffen Brinckmann[1]; Ruth Schwaiger[1];

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Understanding the connections between microstructure and material properties is essential for advancing the development and application of materials in emerging technologies. Traditional two-dimensional (2D) characterization techniques provide limited information on the complex microstructure of materials. In contrast, three-dimensional (3D) characterization offers detailed insights, allowing for a more comprehensive analysis of features such as phase morphologies, spatial distributions, feature volume fractions, and interconnectivity.

This study presents a workflow to construct correlations between microstructure and micromechanical properties based on 3D visualization, focusing on V-Si-B alloys. Automated serial sectioning and two-dimensional optical microstructural stacks were employed for 3D reconstructions and analysis using a fully automated serial sectioning system. A machine learning model, trained on nanoindentation results from a single surface, was applied to all serial sectioning image stacks to construct a 3D hardness map.

The 3D visualization provides valuable insights into how different phases and their spatial arrangement impact the overall material performance, which is beneficial for simulation studies and material design. This approach holds the potential to predict additional microstructure-related properties and can be extended to other material systems.

# Investigation of Hydrogen Segregation at Grain Boundaries in Metals by Combining Ab Initio Calculation and Machine Learning Approach

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Hydrogen segregation at grain boundaries (GBs), which serve as both trapping sites and diffusion pathways, significantly impacts the structural properties of metallic materials. This study investigates how the local atomic environment (LAE) in GBs affects hydrogen segregation behavior by combining *ab initio* calculations and machine learning models. Segregation energies for varying hydrogen concentrations in 20 small alpha iron $\Sigma$ ($\Sigma < 34$) GBs are calculated using density functional theory (DFT) and molecular dynamics (MD) simulations.

A neural network interatomic potential (NNIP), trained by Meng *et al.* [1] for universal Fe-H systems, is validated for hydrogen in iron GBs by comparing the results with DFT calculations. Features for the machine learning models are derived from descriptors such as Voronoi volume, excess length, and the smooth overlap of atomic positions (SOAP) that capture the LAE. The significance of each descriptor is quantified using the permutation importance method. These models are then applied to predict hydrogen segregation energies in high-$\Sigma$ GBs of iron, which are computationally intensive to evaluate with DFT. Furthermore, the transferability of the model to face-centered cubic metallic systems is explored using nickel GBs.

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**Keywords:** hydrogen segregation, grain boundaries, ab-initio calculation, machine learning, local atomic environment

# Useful Information

The symposium will be held at the **Lenthe Auditorium**. It is situated on the first floor of the building at TZA am Europaplatz.
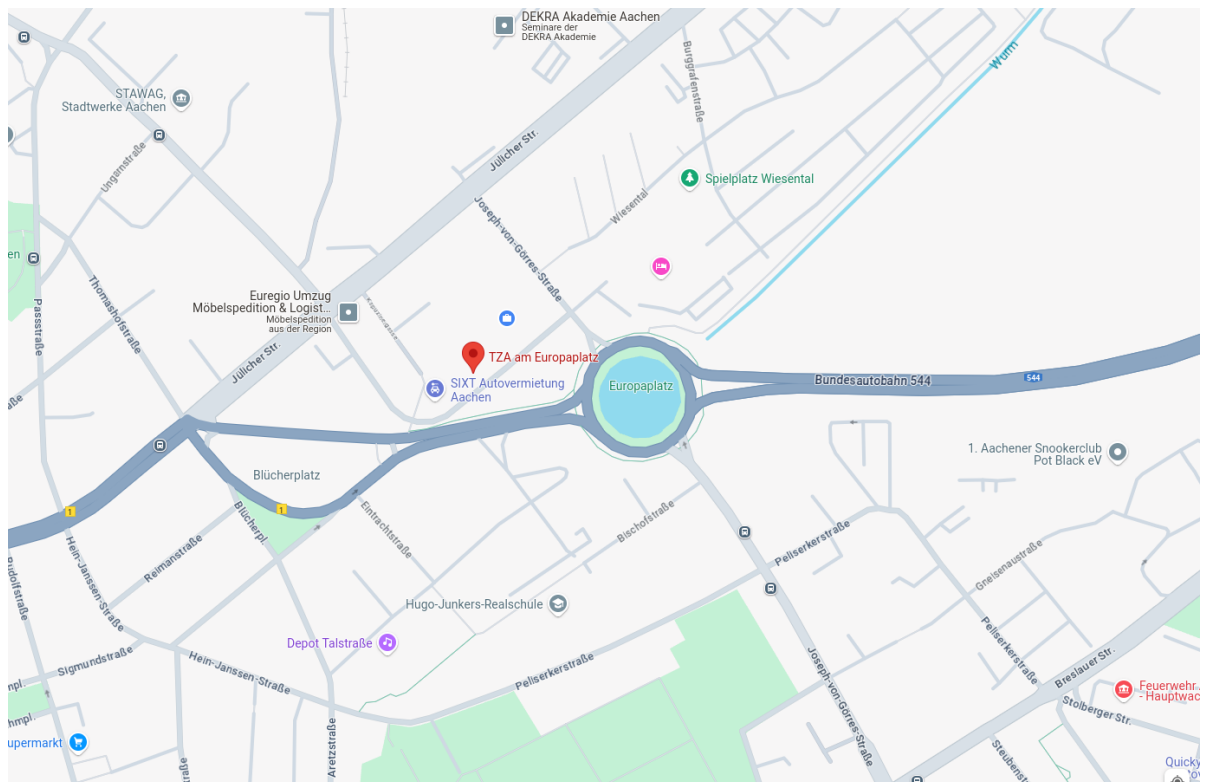
The address is Dennewartstraße 25-27, 52068 Aachen.

Paid street parking is available in front of the building at Dennewartstraße.

Alternatively, there are two parking lots in the close proximity:

1) Blücherplatz

2) APAG Parkplatz am Ludwig Forum

Free wifi is available in the venue without password being necessary. Name of the network: TZA

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# Notes