

Nanobrücken 2025

A Nanomechanical Testing Conference and Bruker User Meeting

March 4-6, 2025 | Fraunhofer IMWS | Halle (Saale), Germany



Preface

Bruker is pleased to announce that **Nanobrücken 2025** will take place March 4-6 in the beautiful city of Halle (Saale).

This is the 15th edition of the Nanobrücken series and combines oral presentations from leading European research groups with practical workshops/tutorials and a poster competition. The conference is open to all aspects of nanomechanical and nanotribological testing, including biomechanical, in-situ experimentation and theory/simulation.

Conference and Bruker User Meeting Venue

Fraunhofer Institute for Microstructure of Materials and Systems IMWS
Walter-Hülse-Straße 1
06120 Halle (Saale), Germany



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Day 1: Tuesday, March 4

Welcome

13:30 Welcome

Jaroslav Lukes, Bruker & Falk Naumann, Fraunhofer IMWS

Talks — Session I

13:45	Correlative in situ micropillar compression for understanding anisotropic hardening in ferrite Angelica Medina, Karlsruhe Institute of Technology (KIT)
14:00	Unraveling structure-process-property relationships of 3D printed polymer microstructures Clara Vazquez Martel, Heidelberg University
14:15	Anti fouling coatings from glassy poly electrolyte multilayers John Akintola, Florida State University
Invited Talk	
14:30	Nanowear resistance of insect mandibles David Labonte, Imperial College London
15:00	Hysitron TI Premier II NanoIndenter David Vodnick, Bruker Nano Surfaces and Metrology

Talks — Session II

15:20

Coffee Break

15:50	Nanomechanical and nanoelectrical analysis of the proton exchange membrane water electrolyzer anode – impact of reinforcement fibers and porous transport layer Julian Borowec, Forschungszentrum Jülich GmbH
16:05	Analysis of thermally activated processes via high temperature scanning indentation Marcel Sos, Technical University of Darmstadt
16:20	Employing shear punch testing to investigate thermomechanical properties of nanocrystalline brass Oliver Petry, Technical University of Darmstadt
16:35	What happens before the first Pop-In? Valeria Lemkova, Saarland University
16:50	Moisture dependence of Polyimide – Silicon Nitride interfaces: comparison between In-Situ and Ex-Situ indentation-induced delamination Filippo Sabatini, STMicroelectronics srl, Politecnico di Milano

Invited Talk

17:05 Mechanistic insights into twinning mechanisms of the Cantor High Entropy Alloy

Christoph Kirchlechner, Karlsruhe Institute of Technology (KIT)

17:35 Welcome Reception & Poster Session

All Posters are Eligible for Top Poster Prize (see list on page 7)

19:30 End of Day 1

Day 2: Wednesday, March 5

Talks - Session III

09:30 Welcome

Oden Warren, Bruker & Erica Lilleodden, Fraunhofer IMWS

Invited Talk

09:40 Elastic microstructures in metallic glasses

Birte Riechers, Federal Institute of Materials Research and Testing (BAM)

10:10 Hysitron PI89 SEM PicoIndenter

Sanjit Bhowmick, Bruker Nano Surfaces and Metrology

10:30 Coupled nanoindentation and EBSD analysis for correlating the microstructure and

mechanical behavior of reduced Iron ore pellets Meriem Ben Haj Slama, Bruker Nano Analytics

10:50 Coffee Break

Talks — Session IV

Invited Talk

11:20 Beyond indentation - the impact of sliding on microstructural evolution

Christian Greiner, Karlsruhe Institute of Technology (KIT)

11:50 Characterization of a damaged bearing steel 100Cr6 using statistical nanoindentation

tests

Romaric Collet, CETIM

12:10 In situTEM nanocompression and nanofriction experiments in vacuum or under water

to investigate the effect of graphene in ceramic composites for tribological applications

Lucile Joly-Pottuz, INSA Lyon

12:30 lce formation and ice friction from laser-patterned spheres

Karlis Gross, Riga Technical University

Lunch Break

12:50 Lunch Provided On-Site

Talks — Session V

Invited Talk 14:00	Nanomechanics in the context of polymer-based composite Thomas Pardoen, Université Catholique de Louvain & WEL Research Institute			
14:30	Application of nanoindentation techniques in the chemical industry: Insights into material performance Svetlana Guriyanova, BASF SE			
14:50	Microscale viscoelastic properties of cement pastes and PMMA quantified by nanoindentation Jiri Nemecek, Czech Technical University in Prague			
15:10	Novel nanoindentation protocol for non-embedded spruce wood and analysis of polymer-modified birch Luis Zelaya-Lainez, TU Wien			
15:30	Coffee Break			
Talks — Session VI				
16:15	Factors that challenge the estimation of the elastic modulus in nanoindenter-loaded monolayer WSe ₂ : A molecular dynamics study Javier Varillas, Czech Academy of Sciences			
16:35	Understanding the fundamentals of grain boundary sliding through micromechanics Divya Sri Bandla, Karlsruhe Institute of Technology (KIT)			
16:55	Mechanisms and anisotropy of serrated flow: insights from microcompression and TEM-based measurements Henry Ovri, Helmholtz-Zentrum Hereon			
Keynote Lecture				
17:15	From nanomechanics of hard phases to microstructure and process design Prof. Dr. Sandra Korte-Kerzel, RWTH Aachen			
18:00	End of Conference Day			
19:00	Conference Dinner			

Day 3: Thursday, March 6

Talks — Session VII

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09:30 Experimental assessment of the mechanical reliability of microelectronics using

advanced micromechanical testing strategies

Andre Clausner, Fraunhofer Institute for Ceramic Technologies and Systems IKTS

10:00 Measurement of interfacial toughness between Polycrystalline and Monocrystalline

Silicon Carbide

Emanuele Cattarinuzzi, STMicroelectronics srl

10:20 Study of epoxy-based molding compound degradation at high temperature operation

using nano-indentation mapping techniques

Falk Naumann, Fraunhofer Institute for Microstructure of Materials and Systems IMWS

10:40 Speeding up Micromechanics Sample Prep for Indentation Testing Using Ultrashort

Laser Pulses

Thomas Höche, Fraunhofer Institute for Microstructure of Materials and Systems IMWS

11:00 Coffee Break

Talks — Session VIII

Invited Talk

11:30 Spherical nanoindentation of nuclear steels

Anna Kareer, University of Oxford

12:00 Nanoindentation - why and when does the tip sharpness matter

Stanislav Zak, Montanuniversität Leoben

12:20 Memorizing the stone age of NI

Matthias Petzold, Fraunhofer Institute for Microstructure of Materials and Systems IMWS

Conference Closing

12:40 Celebrating 50 years of instrumented indentation in Eastern Germany

Lunch

12:50 Lunch Provided On-Site

Demo & Labtour

13:20 PI 89 SEM PicoIndenter demonstration and newTI Premier II demonstration.

Lab tour of IMWS facilities, including: Laserprep, TEM-analytics, Non-destructive

material testing, Micromechanics.



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Poster List

- 1. Investigating the design of macromolecular-based inks for two-photon 3D laser printing Samantha Catt, Heidelberg University
- 2. Nanomechanical studies on ZDDP based tibofilms grown on sapphire substrate Florian Pape, IMKT Leibniz Universität Hannover
- 3. Road to Failure: AFM indentation of polymers Julia Groeger, Erich Schmid Institute
- 4. Determining the elasticity of bacterial cells using microcompression tests Marketa Khyrova, Brno University of Technology
- 5. Investigating the effect of nanoparticles on toughness in silicon nitride thin films Filippo Sabatini, STMicroelectronics srl, Politecnico di Milano
- 6. Viscoelastic Performance of Cellulose Nanofiber-Reinforced Bio-Nanofilms Under Extreme Conditions

Berk Dalkilic, Sinop University

7. Determining the influence of cooling 1.7225 on the microstructural properties using nanoindentation

Matthias Hammes, Leibniz University Hannover, IFUM

8. Quantitative characterisation of the mechanical and electrical properties of nanowires used for nano-energy harvesting

Zhi Li, Physikalisch-Technische Bundesanstalt, PTB

- 9. From atoms to applications: Unraveling coating failure through multi-scale analytical modeling combining molecular dynamics, stress evaluation and surface experiments

 Nick Bierwisch, SIO, Saxonian Institute of Surface Mechanics
- 10. Advanced feedback modes in nanomechanical testing Douglas Stauffer, Bruker Nano Surfaces and Metrology
- 11. In-situ push-to-pull testing of graphene sheets

 Jaroslav Lukes, Bruker Nano Surfaces and Metrology
- **12.** Insights into dispersed mechanical properties

 Ude Hangen, Bruker Nano Surfaces and Metrology
- 13. Study of thermomechanical stability of plasma deposited thin films using in situ high temperature nanoindentation

Vilma Bursikova, Masaryk University

14. Doping strategies to enhance micromechanical strength in sol-gel-derived metal oxide semiconductors

Seydanur Kaya, Kastamonu University

- 15. Surface Investigation by Atomic Force Microscopy of a Li-ion battery electrode Monika Parihar, Université Paris Saclay
- 16. Stability of nanoparticles with a focus on their morphology, and mechanical properties with the dynamics of proteins as well as other biomolecules for health diseases Seniha Simale Su Uygan
- 17. Predicting the microstructural evolution of ion-irradiated Eurofer97: Nanoindentation study supported by CPFEM and TEM

Tymofii Khvan, National Center for Nuclear Research (NCBJ)



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Abstracts - Oral Presentations

Day 1: Tuesday, March 4th

Talks — Session I

13:45 Correlative in situ micropillar compression for understanding anisotropic hardening in ferrite

Angelica Medina¹, Divya Sri Bandla¹, Subin Lee¹, Christoph Kirchlechner¹, Reinhard Pippan²

1 Institute for Applied Materials, Karlsruhe Institute of Technology, D-76344 Eggenstein-Leopoldshafen, Germany

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Automotive structural components frequently employ dual-phase (DP) steels, renowned for their exceptional strength and ductility. Because of their soft ferrite matrix and scattered hard martensite islands, DP steels have a complex microstructure that requires a full understanding of their mechanical behavior to maximize manufacturing efficiency and enhance damage tolerance. Previous studies have reported the strain-hardening capability of ferrite and its role in suppressing nucleation and void growth [1]. However, traditional isotropic strain hardening assumptions have limitations, such as the inability to describe the dependence of the load path on hardening and, consequently, are not suitable for load path optimization in representative solid elements.

In this presentation, we present the study on ferrite's anisotropic strain hardening capabilities in DP800, which suppress damage growth during load path changes. In situ scanning electron microscope (SEM) micropillar compression tests were conducted together with detailed dislocation analysis via controlled electron channeling contrast imaging (cECCI).

To control the dislocation density and their slip systems, multiple samples with different dislocation structures were prepared from a single macroscopic test of the region near the Lüders bands, where the dislocation structure changes drastically. Next, we located grains where the possibility of finding a single slip is maximized and subsequently directly correlated the critical resolved shear stress (CRSS) along with the local characteristics of dislocations, including their Burgers vector, slip systems, and local dislocation density, in a high-throughput manner to understand the latent hardening behavior of ferrite. It is believed that this study will serve as a micromechanical basis for damage-controlled forming techniques and will be essential for predicting damage evolution.

[1]https://doi.org/10.1016/j.matdes.2024.112630

14:00 Unraveling structure-process-property relationships of 3D printed polymer microstructures

Clara Vazquez-Martel*1, Samantha O. Catt1, Eva Blasco1

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- * clara.vazquez@oci.uni-heidelberg.de

Additive manufacturing, also known as 3D printing, is revolutionizing academia and industry as an intelligent fabrication tool. Notably, two-photon 3D laser printing enables precise micro- and nanoscale fabrication, offering potential applications across various fields, including optics, biomedicine, and soft robotics. The properties of printed microstructures are determined by their chemical structure and formulation, as well

as many 3D printing process parameters. Understanding the behavior of these 3D printed microstructures is essential for their successful integration into real-world applications. As research in novel high-resolution and functional materials increases, there is an urgent demand to control their (macro)molecular architecture and to understand structure-process-property relationships through systematic characterization. Mechanical characterization at microscopic scales is challenging, and little data for some commercially available materials is available in the literature. Furthermore, the lack of standardized procedures for characterizing mechanical properties at small scales, particularly for soft materials like polymers and hydrogels, results in inconsistent data collection and analysis, complicating the comparison of results across studies. To address this, we leverage our expertise in designing and synthesizing (macro)molecularly defined printable materials to develop a comprehensive framework for characterizing 3D printed polymer microstructures spanning a wide range of mechanical properties from kPa to GPa. In recent studies, we have utilized nanoindentation to establish an accessible database as a framework toward advanced materials and microstructures with tailored mechanical properties for various 3D printing applications.

14:15 Anti fouling coatings from glassy poly electrolyte multilayers

<u>John Akintola*</u>, Yuhui Chen, Zachary Digby and Joseph Schlenoff Florida State University

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Coatings that prevent or decrease fouling are sought for many applications, including those that inhibit the attachment of organisms in aquatic environments. To date, antifouling coatings have mostly followed design criteria assembled over decades: surfaces should be well/strongly hydrated, possess low net charge, and maintain a hydrophilic character when exposed to the location of use. Thus, polymers based on ethylene glycol or zwitterionic repeat units have been shown to be highly effective. Unfortunately, hydrated materials can be quite soft, limiting their use in some environments. In a major paradigm shift, this work describes glassy antifouling films made from certain complexes of positive and negative polyelectrolytes. The dense network of electrostatic interactions yields tough materials below the glass transition temperature, Tg, in normal use, while the highly ionic character of these polyelectrolyte complexes ensures strong hydration. The proximity of equal numbers of opposite charges within these complexes mimics zwitterionic structures. Films, assembled layer-by-layer from aqueous solutions, contained sulfonated poly(ether ether ketone), SPEEK, a rigid polyelectrolyte that binds strongly to a selection of quaternary ammonium polycations. Layer-by-layer buildup of SPEEK and polycations was linear, indicating strong complexes between polyelectrolytes.

Invited Talk

14:30 Nanowear resistance of insect mandibles

David Labonte

Department of Bioengineering, Imperial College London, UK

Many biological materials have remarkable properties, and may form key components of fitness. In this talk, I will discuss three examples of our work on the nanomechanical properties of biological materials: (1) Leaf-cutter ants cut plant matter on an almost industrial scale - about every 5th leaf in the Neotropics falls victim to their relentless attacks. Plants, of course, attempt to defend themselves, and the result is an evolutionary arms race in which ants evolved adaptations to minimise wear of their razorsharp mandible cutting edges, and plants evolved ways to maximise it. How wear resistant are leaf-cutter ant mandibles? (2) Insect herbivores more generally have enormous ecological and economic impact on our lives - they act as ecosystem engineers through the cycling of nutrients, and consume almost every 6th crop calorie destined for human consumption. A core component of this process is the mechanical processing of food, facilitated by the "design" of the insect head capsule. Using high-throughput indentation techniques, we mapped the mechanical properties across the head of a notorious insect pest, with the ultimate aim to understand the evolution of insect herbivore head form. (3) Insects aren't the only animals who have to worry about their teeth. Komodo dragons are the largest extant predatory lizards, and possess a tooth type that makes them

valuable analogues for studying tooth structure and function in theropod dinosaurs. We revealed that V. komodoensis teeth possess a unique adaptation: iron-enriched coatings on their tooth serrations and tips enhance tooth mechanical properties, and so help keeping tooth cutting edges sharp.

15:00 HysitronTl Premier II NanoIndenter

David Vodnick

Bruker Nano Surfaces and Metrology

Bruker's next-generation TI Premier II nanoindenter is the latest addition to our family of industry-leading Hysitron testing systems. The new TI Premier II offers a more accessible solution for comprehensive environmental control including heating to 800°C, cooling to -120°C, and combined humidity & temperature control. This new expanded capability on our popular, multi-user nanoindenter platform can all be achieved in combination with advanced testing modes such as: XPM high-speed indentation mapping at 4 indents per second, in-situ SPM imaging for targeting and post-indent location verification, continuous measurement of hardness & modulus via Dynamic nanoindentation, and nano-scratch testing. TI Premier II therefore provides extensive value with best-in-class comprehensive nanomechanical testing. The TI Premier II base configuration offers a suite of out-of-the-box testing and analysis capabilities for precise nano- to microscale characterization, and easy sample mounting with the redesigned universal sample mounting stage. A modular design also makes the system easily upgradable at any time to accommodate specialized testing methods and environmental control. Only Bruker's newTI Premier II combines leading sensitivity and adaptability with automated efficiency: •Advanced capacitive transducer technology ensures the most accurate and repeatable measurements. •Best-in-class range of testing techniques and sample compatibility simplifies experiment design. •New TriboScan 12 control software and integrated environmental isolation streamlines testing and guarantees reliable results, especially with the Tribo iQ data analysis toolkit provided.

Talks — Session II

15:50 Nanomechanical and nanoelectrical analysis of the proton exchange membrane water electrolyzer anode – impact of reinforcement fibers and porous transport layer

<u>Julian Borowec</u> ^{12*}, Lukas Rein ¹², Nelli Gorin ¹², Shibabrata Basak ¹, Ladislaus Dobrenizki ³, Günter Schmid ³, Eva Jodat ¹, André Karl ¹, Rüdiger-A. Eichel ¹²⁴, Florian Hausen ¹²

- 1 Institute of Energy Technologies Fundamental Electrochemistry (IET-1), Forschungszentrum Jülich GmbH, 52425 Jülich, Germany,
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- 3 SETI SES PRM (Sustainable Energy Solutions Product Management), Siemens Energy Global GmbH~\&~Co. KG, 91058 Erlangen, Germany
- 4 Faculty of Mechanical Engineering, RWTH Aachen University, 52062 Aachen, Germany.
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Understanding the degradation of proton exchange membrane electrolyzer cells (PEMECs) is critical for durability improvements. In this work,[1] a large-scale web-woven reinforced membrane electrode assembly (MEA) anode, was long-term operated (>5000 hours) and analyzed by nanomechanical and nanoelectrical atomic force microscopy (AFM) techniques and nanoindentation. The web-woven fibers were found to locally enhance the reduced modulus and hardness, making them an effective reinforcement for extended operation. Notably, both pristine and operated anodes exhibited slightly reduced electrically conductive surface areas at intersections of reinforcement fibers. While the pristine anode was initially homogeneous, it heterogenized upon operation, showing additional domains related to the porous transport layer (PTL) and increased statistical deviations. Nanoindentation revealed an increased reduced modulus and hardness upon operation, accompanied by a near surface stiffening of the catalyst shown by AFM. This effect is promoted by the loss of low-stiffness ionomer. Confirmed by the increase of electrically conductive anode surface area. The most

pronounced aging effects were observed only at a small fraction of the surface, particularly at specific PTL-related features. This study provides the first detailed analysis of a web-woven fiber-reinforced MEA, offering new insights into anode aging mechanisms associated with reinforcement fibers and PTL.

[1] Borowec, Julian, et al. ""Nanomechanical and Nanoelectrical Analysis of the Proton Exchange Membrane Water Electrolyzer Anode—Impact of Reinforcement Fibers and Porous Transport Layer." "Journal of Materials Chemistry A (2025). DOI: 10.1039/D4TA07367C

16:05 Analysis of thermally activated processes via high temperature scanning indentation

<u>Marcel Sos*</u>, Sebastian Bruns, Enrico Bruder, Karsten Durst Physical Metallurgy (PhM), Department of Materials and Earth Sciences, TU Darmstadt * marcel.sos@tu-darmstadt.de

Nanoindentation serves as a tool to map mechanical properties of materials not only at room temperature, but also at temperatures at or close to the operating temperature of materials used for high temperature applications. Thermally activated processes can then be characterized via the calculation of strain rate sensitivity and activation volume at different temperatures.

Testing at elevated temperatures, however, presents its own issues in the form of thermal drift due to temperature mismatch and pronounced tip-sample-interactions.

The temperature matching procedure in particular can be time-consuming, which limits the applicability of conventional high temperature nanoindentation to materials with sufficiently stable microstructures at the testing temperature. To overcome some of these limitations, the High Temperature Scanning Indentation (HTSI) technique was introduced by Tiphene et al. [1]. It utilizes a large amount of high speed tests (on the order of 1s each) to generate a high density of data points and measure mechanical properties during thermal ramping of a sample. This allows for not only the investigation of dynamic processes like recrystallization, but also for the characterization of brittle-to-ductile or phase transitions with unprecedented data density.

In this contribution, the HTSI technique is applied to investigate the thermal stability of ultrafine-grained copper alloys processed via high pressure torsion [2,3], following different thermal profiles to evaluate hardness and strain rate sensitivity of a function of temperature and time. Fused silica is used as a reference material to show the reliability of the obtained data and the comparability to other testing methods. Additionally, the applicability to brittle-to-ductile transitions is demonstrated on Chromium.

[1] G. Tiphene J. Mater. Res. 2021, 36(12), 2383-2396

[2] E. Bruder Scripta Mater. 2018, 144, 5-8

[3] T. Keil IOP Conf. Ser.: Mater. Sci. Eng. 2022, 1249(1), 12003"

16:20 Employing shear punch testing to investigate thermomechanical properties of nanocrystalline brass

Oliver Petry*, Karsten Durst*, Sebastian Bruns*, Mareike Seitel*, Naeimeh Fakhar*

- * Physical Metallurgy, Department of Materials and Earth Sciences Technical University of Darmstadt, Darmstadt, 64287, Darmstadt, Germany
- + Mechanical Engineering Department, Hamedan University of Technology, 65155-579 Hamedan, Iran.
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The shear-punch test can be used to evaluate the mechanical properties of materials with limited availability. The shear-punch test was used to evaluate ultimate shear strength and shear yield strength of nanocrystalline (nc) brass (Cu-30 wt% Zn) and a linear relation between ultimate strength and yield strength from uniaxial compression and tensile tests could be confirmed. A softening could be observed at high strains, and was investigated for different temperatures (RT, 100°C, 200°C, 300°C). The softening was related to a change in geometry and a method to evaluate an effective thickness from laser-microscopic images was established. Strain-rate-jump (SRJ) tests were employed using the shear punch method and the results were compared to uniaxial compression and nanoindentation SRJ tests. The results show the breakdown of the Hall-Patch effect

in nc brass at elevated temperatures and good agreement of strain rate exponents calculated from tensile, compression and nanoindentation tests. Furthermore, the SPT allows the investigation of the microstructure in highly deformed area and undeformed area, showing stress assisted grain growth after testing at 200 °C.

16:35 What happens before the first Pop-In?

Valeria Lemkova* 12, Christian Motz 1, Ralf Busch 2, Florian Schäfer 1

- 1 Institut for Material Science and Methods 2 Institut for Metallic Materials
- * v.lemkova@matsci.uni-saarland.de

Plastic deformation behavior is important in the field of amorphous metals as it determines the limits of possible applications. Understanding the onset of plasticity is essential, and micromechanical tests, such as nanoindentation, offer a unique methodology to locally study deformation processes with a resolution of just a few nanometres. To investigate the plastic behaviour of amorphous metals, systematic nanoindentation studies were performed on different alloys, using indentation depths of up to 50 nanometres. Systematic nanoindentation studies up to 50 nanometers have been carried out on various alloys. The aim of the present study is to separate the evolution of serration from a true elastic response using spherical nanoindentation studies on the scale of only a few nanometers indentation depth. Our results reveal that the onset of plasticity occurs before the first pop-in, suggesting that the elastic-plastic transition in BMGs is not abrupt but continuous. This implies that the commonly accepted understanding of the inception of plastic deformation in amorphous metals, namely the nucleation of shear transformation zones, seems to be incomplete and needs further attention.

16:50 Moisture dependence of Polyimide – Silicon Nitride interfaces: comparison between In-Situ and Ex-Situ indentation-induced delamination

<u>Filippo Sabatini</u>, Emanuele Cattarinuzzi, Vincent Coutellier, Paola Zuliani, Andrea Li Bassi Politecnico di Milano, STMicroelectronics

In the framework of the 300 mm transition in the semiconductor industry, several new challenges have emerged from the introduction of new materials and modified structures. In this context it has been necessary to validate a new stack of protective layers for a power device composed by a polybenzoxazole (PBO) molding compound and a silicon nitride (SiN) passivation layer. PBO adhesion was evaluated on different substrates, varying SiN stoichiometry and applying two different plasma surface treatment. In addition, the influence of temperature (T) and relative humidity (RH) on adhesion was tested comparing two approaches: classical aging (48 hours and 7 days in a climatic chamber) followed by nanoindentation (ex-situ testing) and nanoindentation under controlled conditions using Bruker's Tl980 xSol | Humid module (in-situ testing). Nanoindentation was used to evaluate the critical load for delamination: results revealed a dependence of PBO adhesion on the environmental dew point. Moreover, a more significant degradation of adhesion was observed during insitu testing, suggesting a partial recovery of adhesion after aging. These findings highlight that post-aging adhesion measurements may not fully reproduce real-world environmental conditions, underlining the importance of in-situ analysis for accurate adhesion assessment.

Invited Talk

17:05 Mechanistic insights into twinning mechanisms of the Cantor High Entropy Alloy

Christoph Kirchlechner

Karlsruhe Institute of Technology (KIT)

The Cantor alloy, a prominent first-generation high-entropy alloy, was first introduced in 2004 and exhibits increased strength and ductility at cryogenic temperatures, a behavior distinct from other alloys. The plastic deformation in this CoCrFeMnNi alloy is primarily governed by twinning. This study explores the role of

the short-range ordering and stacking fault energy, by studying its microstructure changes along with its microscale mechanical response.

Abstracts - Oral Presentations

Day 2: Wednesday, March 5th

Talks — Session III

Invited Talk

09:40 Elastic microstructures in metallic glasses

Birte Riechers

Federal Institute of Materials Research and Testing (BAM), Unter den Eichen 87, 12205, Berlin, Germany

Metallic glasses have a disordered atomic structure and are known to be heterogenous materials in both space and time, where temporal time scales are intimately linked to structural length scales. Not only structural relaxation times span orders of magnitude, but there is now compelling evidence from experiments that also structural fluctuations, probed via nano-mechanical methods, may span across scales. Primarily revealed in the few nm-regime, we here set out to understand the origin of elastic fluctuations at much larger scales. We demonstrate the existence of an elastic decorrelation length of the order of 100 nm in a Zr-based bulk metallic glass using spatially resolved elastic property mapping via nanoindentation.

As compositional modulations sufficiently large to account for this elastic microstructure were not identified, we argue that the revealed long-range elastic modulations stem from structural variations affecting the local density. Here, high-throughput nano-indentation proves as a powerful tool for the characterization of processing-induced property fluctuations and bears the potential of formulating structure-property relationships for metallic glasses.

10:10 Correlation of Mechanical Properties, Microstructure, and Analytical Mapping of Materials using PI 89 Auto SEM PicoIndenter

<u>Sanjit Bhowmick</u>, Kevin M. Schmalbach, Eric D. Hintsala, Douglas D. Stauffer Bruker, Minneapolis, MN, USA.

The Hysitron PI 89 Auto SEM PicoIndenter offers several advantages for in situ nanomechanical testing, including automated testing capabilities and correlative microscopy integration. The PI 89 Auto support colocalized acquisition of in-situ mechanical data at user defined regions of interest from imaging and analytical mapping. This study highlights the use of a dual-axis rotation and tilt (R/T) stage with the PI 89 PicoIndenter for advanced micro- and nano-mechanical testing. The first case study examines microscale fracture in single-crystal silicon using wedge-loaded double cantilever beams. The dual-axis R/T stage ensures precise alignment of the notched specimen with the wedge probe, facilitating mode I loading. Stable crack growth along a {111} plane produces characteristic hackle and arrest lines, enabling the determination of a crack resistance curve [1]. The second case study focuses on correlative nanoindentation and electron microscopy for microstructural characterization. The R/T stage enables precise alignment in the SEM for energy-dispersive spectroscopy (EDS), electron backscatter diffraction (EBSD), and nanoindentation. This method is applied to laser-clad steel, demonstrating a strong correlation between hardness and microstructure. The dual-axis R/T stage proves invaluable for both fracture mechanics and correlative nanoindentation studies. These findings underscore its importance in achieving precise specimen alignment, ensuring reliable and accurate mechanical property measurements.

[1] F.W. Delrio et al, Materials Research Letters 10 (2022) 728–735.

10:30 Coupled nanoindentation and EBSD analysis for correlating the microstructure and mechanical behavior of reduced Iron ore pellets

Meriem Ben Haj Slama* 1, Heidi Bögershausen 2, Ude Dirk Hangen3

- 1)Bruker Nano GmbH _ Electron Microscope Analyzers
- 2)Max Planck Institut for Sustainable Materials GmbH
- 3) Bruker Nano Surfaces and Metrology Division"

Hydrogen direct reduction (HyDR) of iron ore pellets is an environment-friendly alternative for steel making compared to carbon reduction procedures. In HyDR, hydrogen diffuses fast through ore pellets, but the kinetics of iron-oxide transformation into iron are still not fast enough for high-quantity steel production. Thus, understanding the mechanisms that control the reduction kinetics is a key step towards speeding them up. These kinetics depend partially on the microstructural features (such as interfaces, cracks, pores, and phase distribution).

In the aim of providing more insight into these micro-scale properties and the mechanisms involved in the HyDR transformations, the present work correlates hardness and reduced modulus maps with phase/crystal orientation/defect maps of three iron pellets at different HyDR stages (non-reduced, partially reduced and fully reduced). For this approach, Electron Backscattered Diffraction (EBSD) analyses are coupled with nanoindentation mappings of the same areas of interest.

The results indicate a fine sub-micron structure for both non-reduced and partially reduced samples. Such a structure necessitates high-quality sample preparation, which is challenging due to the sample's porosity. Despite these experimental challenges, there is a very good agreement between the phase analysis by EBSD and the nanoscale hardness measurements. EBSD allows direct measurement of different phase distributions and crystal orientations. Hardness measurements differentiate between soft metallic and hard mineral phases, enabling the observation of iron oxides versus the reduced iron with high spatial resolution. Both hardness tests and EBSD analysis show a perfect match for the different phases even for tiny amounts of these phases. The correlation of the two independent approaches evidenced small amounts of oxide in the ferrite matrix of partially reduced sample, far from being a simple effect of measurement uncertainties.

Talks — Session IV

Invited Talk

11:20 Beyond indentation - the impact of sliding on microstructural evolution

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Tribological contacts are almost everywhere and arise on all length scales. In metals, tribological loading result into a deformation layer in which wear particles are initiated. Increasing the life time of parts under tribological loading therefore requires fundamental understanding of the determining microstructural processes. To this end, a tribological model system was chosen consisting of single-crystalline copper paired against sapphire spheres sliding in a dry fashion. To demonstrate the impact of sliding on these elementary processes, results of a single trace tribological experiment are compared to (nano) indentation. Doing so highlights differences concerning slip traces and crystal rotation. Next, the influence of crystallographic orientation is presented. Here, a universal crystal rotation around the transversal axis with respect to the sliding direction was uncovered. Differences in topography and microstructural characteristics were observed

with in- and off-symmetry crystallographic orientations (e.g. ND[110]/SD[001] vs ND[110]/SD[11-2]). To explain these differences in microstructural evolution, a stress field model was developed allowing to calculate the resolved shear stresses on each slip system. In order to experimentally validate these calculations, we made use of deformation twinning, as twins allow to probe the stress field. Multiple stress field assumptions are evaluated: very simplified single stress approaches, a stress field based on Hertzian contact mechanics and stress fields calculated with finite element methods using isotropic material models as well as applying crystal plasticity. We found that an isotropic material model considering plasticity results into a significant improvement in prediction quality compared to more traditional solutions.

11:50 Characterization of a damaged bearing steel 100Cr6 using statistical nanoindentation tests

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The damage suffered by bearings due to the reciprocating movements of small amplitudes has been not yet sufficiently controlled or studied. This form of stress at low amplitudes is commonly referred to as fretting, but in the context of this study involving bearings, it is more aptly termed false Brinelling. The main objective of this work is to understand the mechanisms engaged during "false brinelling" and identify the evolution of the material according to the severity of the false brinelling. The presented study is a material characterisation using micromechanical tests to highlight and understand the degradation mode engaged during false brinelling. Nanoindentation tests were performed to identify an eventual modification of the mechanical response within the material related to deformation due to false brinelling mechanisms. 100Cr6 steel is widely used as bearing, for this reason this material was investigated. Because of its microstructure (martensite with cementite precipitates), such material shows a very scattered mechanical response and the identification of a local modification of the mechanical properties related to false brinelling using a 100Cr6 is a true challenge.

First, mechanical response of both the cementite precipitates and the martensite were investigated using SPM with appropriate polishing steps. Then, statistical analyses were performed at different areas within different samples (showing different state of degradation). Nanoindentation (and statistical analyses) appears to be a way to characterize relatively small modification of the mechanical response even through the modification is lower than the natural dispersion of the material. Others material characterizations were performed (such as EBSD &TEM) that are consistent with the nanoindentation results.

12:10 In situ TEM nanocompression and nanofriction experiments in vacuum or under water

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Key Words: TEM, in situ, friction, compression, ceramics

Friction is probably one of the most important challenges for the scientific and industrial communities because it directly impacts the energy efficiency of mechanical systems. Indeed, a considerable amount of energy is wasted to overcome friction, especially in transport, industrial and power-generation sectors, and major economic losses are also due to wear of components and their replacement. Few-Layered Graphene (FLG) ceramic-matrix composites containing a high-quality graphene dispersion providing remarkable improvements in both mechanical, functional properties [1,2] and good compatibility with environmentally-friendly water-based lubricants could be a breakthrough technological solution.

FLG-Si₃N₄ composite powders were prepared, without the manipulation of any pre-existing graphite or graphene platelets, by a method using the chemical vapor deposition of carbon (using CH4 as the carbon gas) onto the particles of a commercial Si₃N₄ powder [3]. The composite samples densified by Spark Plasma Sintering were investigated for dry- or water-lubricated tribological applications. Results obtained from macroscopic friction tests show that the increase of the number of layers in the FLG stack, typically from 4 to 35, leads to a significant improvement in tribological properties, especially in water-lubricated conditions where ultralow friction (friction coefficient < 0.01) was achieved with no significant wear. To better understand the tribological properties observed, H-bar samples were prepared by FIB and tested in situ in an Environmental Transmission Electron Microscope. The influence of the graphene layers was investigated in compression and under friction, as well as that of the environment (under vacuum or with water vapor). TEM observation conditions have been optimized, as well as image processing to follow deformation phenomena and the impact of the graphene on the mechanical properties of the composites.

- [1] P. Miranzo, J. Eur. Ceram. Soc. 37 (2017), 3649.
- [2] K. Markandan, J. Mater. Res. 32 (2017), 84.

deforms the ice over which sliding occurs.

- (3] A. Berrais, J. Eur. Ceram. Soc. 45 (2025), 116799.
- [4] The authors acknowledge the Consortium Lyon Saint-Etienne de Microscopie (CLYM) for the access to the microscope. The work was carried out as part of the ANR project "GReC" (ANR-20-CE08-0008-03).

12:30 Ice formation and ice friction from laser-patterned spheres

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Testing in extreme environments is becoming increasingly important due to the rise in space travel activities and the growing interest in winter sports. Here we shall focus on ice-friction for winter sports. Before determining ice-friction it is necessary to calibrate a large probe, form ice and prepare a flat ice-surface. All these steps require new considerations and approaches that are highly specific to testing at sub-zero temperature conditions. Furthermore, we will laser pattern spherical probes and slide them across ice to simulate sliders used in ice sports, with the expectation that the patterns on the slider surface will affect the ice friction. Results will show a) a new way of performing the tip-to-stage calibration, b) ice formation from an exotherm during cooling as well as a video to show microstruture formation, c) different attempted methods for in-situ ice surfacing, and d) the ice friction measurements from differently patterned spheres. Ice friction, generally thought to be a surface phenomenon, will be shown to combine the effects of surface sliding resistance and plastic deformation, which

Talks — Session V

Invited Talk

14:00 Nanomechanics in the context of polymer-based composite

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Constitutive models for thermoplastics and thermosets have been enhanced for more than 30 years to account for a range of phenomena including hardening-softening-re-hardening, viscoplastic effects, back stress upon unloading, and aging-rejuvenation. These models offer good predictive capabilities, although with a very large number of parameters. However, predictions are unsatisfactory when these models are used to simulate, after identification on bulk data, the response of composites. Indeed, direct simulations of UD RVE tend to underestimate the load carrying capacity. Comparison of digital image correlation (DIC) data with FE simulations at the level of a UD RVE also show discrepancies between predicted and experimental strain maps. Nanoindentation in matrix pockets between fibers confirm that local strength is larger than bulk. All this indicates that the polymer when confined by fibers does not behave as in bulk form. So, either the constitutive model is not rich enough and should include size effects to capture local confinement and large plastic strain gradients, or the polymer nature is locally different, or both effects play a role. In this context, nanomechanical tests were developed to probe the near fiber matrix response: compression tests in SEM coupled to nano-DIC with resolution below 100nm, AFM modulus mapping, and nanoindentation. This was applied to thermoset / carbon fiber and thermoplastic / glass fiber systems. Interphase region of couple of hundreds nm is detected. Fine localization band patterns are resolved that cannot be predicted by FE with conventional models. Nanoindentation reveals hardness changes with indentation depth pointing towards size dependent plasticity. A micromorphic gradient plasticity model has been applied to fiber matrix representative cells showing excellent capabilities to capture both adequate strain fields as well as the size induced strengthening.

14:30 Application of Nanoindentation Techniques in the Chemical Industry: Insights into Material Performance

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In the rapidly evolving landscape of the chemical industry, the need for precise characterization of materials is of great importance. This presentation demonstrates a comprehensive examination of the application of nanoindentation techniques across various materials commonly utilized in the sector, including polymer materials, blends, 3D printed constructs, coatings, and battery materials. By employing both quasistatic and high-speed nanoindentation, we derive critical mechanical parameters such as hardness, elastic modulus profiles, and detailed mapping of material properties. These measurements not only elucidate the mechanical behavior of the tested materials but also provide a comparative analysis with elastic modulus results obtained through Atomic Force Microscopy (AFM) in the PeakForce-Quantitative Nanomechanical (PF-QNM) mode, thereby validating our findings. Furthermore, we explore the utility of scratch testing via nanoindentation to estimate critical load thresholds that signify material failure, offering insights into the durability and wear resistance of coatings. Additionally, the phenomenon of particle fracture is investigated through targeted nanoindentation, allowing for a deeper understanding of the mechanical integrity of battery materials and their performance under stress. This multifaceted approach underscores the relevance of nanoindentation techniques

in enhancing the material selection and design processes in the chemical industry, ultimately leading to improved performance and longevity of products.

14:50 Microscale viscoelastic properties of cement pastes and PMMA quantified by nanoindentation

J. Nemecek*, J. Nemecková, J. Nemecek

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Long-term deformations such as creep, which appear on bridges and other structures subjected to permanent loads, typically show a logarithmic or power-law trend. Similar dependencies can be observed even in shorter loading times with the help of nanoindentation tests, when a sharp or blunt tip is pressed into the material. Although the creep trends are similar, the deformation under the tip causes inelastic deformations, which must be separated from purely viscoelastic ones. This contribution studies the effect of indenter shape, loading speed and evaluation procedure on viscoelastic properties, filtering out undesirable plastic strains and the linearity of creep on cement pastes. Results for cement pastes are verified on a reference polymeric material, PMMA, whose trends in creep evaluation from nanoindentation tests pose a similarity.

15:10 Novel Nanoindentation Protocol for Non-Embedded Spruce Wood and Analysis of Polymer-Modified Birch

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This study presents two nanoindentation campaigns aimed at investigating the micromechanical properties of wood. A novel milling, polishing, and indentation protocol was developed to analyze non-embedded Norway spruce, eliminating the need for resin embedding, which can alter mechanical properties. The protocol demonstrated reliable results, showing reduced modulus and hardness values of approximately 12% and 26% lower, respectively than those reported for embedded spruce in the literature. These findings align with data from Loblolly pine, validating the method's robustness.

The second campaign focused on polymer-modified, delignified birch to evaluate how different polymers influence stiffness. Nanoindentation results revealed a significant dependency of micromechanical properties on the polymer type used for modification. This new novel material is intended to be used as a greener option to traditional CO2-intensive glass.

Overall, these findings highlight the critical role of micromechanical characterization in understanding and optimizing wood materials. The developed protocol and insights into polymer effects provide valuable knowledge for advancing wood-based products with enhanced performance characteristics.

Talks — Session VI

16:15 Factors that challenge the estimation of the elastic modulus in nanoindenter-loaded monolayer WSe2: A molecular dynamics study

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We performed large-scale molecular dynamics (MD) simulations to investigate the nanoindentation response of suspended monolayer WSe2. The construction of the MD systems considers various settings that mimic those commonly found in membrane nanoindentation testing, in which 2D samples are supported on substrates featuring circular holes of diameter D, and indentation loading is exerted at the hole's center where the 2D material is freestanding. In our nanoindentation simulations, displacement-controlled, low-load indentation is applied by an Au indenter tip of radius R<<D. MD indentations using a rigid nanoindenter and a sufficiently large clamped circular WSe2 membrane (D≈30R) produce a mechanical response that matches the conditions assumed for the analytical derivation of the solution for the point-loaded circular membrane. Under these conditions, the elastic modulus for monolayer WSe2 is E≈188 MPa. The E values were extracted from the indentation force, (F)-indenter displacement (δ) curve in the stretching-dominated regime where F follows a cubic relation with δ. We show that the attainment of the cubic regime is attained when the membrane is clamped at the substrate hole's edge, regarding sliding processes of the membrane over the substrate as negligible. This is an assumption often difficult to make in mechanical testing of 2D materials. Deviations from such conditions can lead to mechanical responses that produce nontrivial misestimations of E mainly attributed to plastic deformations in the indenter tip, flexible substrates, nonlinear elastic deformation, and/or non-clamped membrane-substrate boundaries. These results highlight the importance of ensuring that boundary conditions and geometries align with those assumed in the theory providing the analytical solution. Accurate mechanical characterization is crucial for the application of 2D materials as ultrasonic transducers. J.V. acknowledges financial support from the ERA-NetTH80020003 project.

16:35 Understanding the fundamentals of grain boundary sliding through micromechanics

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One of the challenging deformation behaviors of polycrystalline materials to understand is grain boundary sliding. The inevitable microstructural features like triple junctions and a variety of grain boundaries make it difficult to study the fundamental aspects of sliding of the single grain boundary. In situ scanning electron microscope (SEM) micropillar compression testing can overcome this challenge by testing micropillars containing a single grain boundary.

It is well-known that grain size plays an essential role in polycrystalline materials' grain boundary siding behavior. Does it hold the same for bicrystals, where essentially only one grain boundary exists? To study the fundamental differences between the sliding behavior of polycrystal and bicrystal, nickel (Ni) was chosen as the model material. Micropillars of diameter ranging from 0.5 to 6 µm were prepared using focused ion beam milling. All the micropillars contain two grains (normal orientations are close to [324] and [344]) with a random

high angle grain boundary (\sim 20°) that runs through the pillar and is inclined to the compression axis at an angle of \sim 35°. The micropillar compression tests at different temperatures revealed a change in deformation behavior from slip domination to grain boundary sliding domination (at least 40 to 65%) at a temperature of 573 K and strain rates of 10-3 and 10-2 s-1. These grain boundary sliding conditions were insensitive to the pillar diameters ranging from 0.5 to 6 μ m. However, the critical shear stress to activate the sliding was sensitive to the pillar diameter; smaller pillar diameters required more stress. This observation supports the dislocation-mediated sliding mechanism rather than the diffusion-mediated mechanism. Compared to the polycrystalline data from the literature, the current study showed grain boundary sliding at lower temperatures. The origin of this behavior will be discussed during the talk.

16:55 Mechanisms and anisotropy of serrated flow: insights from microcompression and TEM-based measurements

Henry Ovri

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Serrated flow is commonly observed across length scales that span the macro-, meso- and submicron-scales. However, the phenomenon has been primarily studied at the macron scale. As a result, critical aspects such as the influence of plastic anisotropy and twinning are yet to be understood. Besides, establishing direct correlations between macroscopic manifestations of serrated and the underlying micromechanisms is essential. Bridging these gaps requires combinatory microcompression of single crystals and TEM based measurement. These techniques were employed in this work on pure Mg and a Mg-Gd alloy single crystals, oriented for twinning and slip on different planes. The work clearly demonstrates that serrated flow is strongly anisotropic. I will highlight the mechanistic origin of the anisotropy and the implications for ductility in Mg-RE based alloys.

Keynote Lecture

17:15 From nanomechanics of hard phases to microstructure and process design

Martina Freund, Christina Gasper, Zhuocheng Xie, Tobias Stollenwerk, Nisa Ulumuddin, Pei-Ling Sun, Maximilian Wollenweber, Setareh Medghalchi, Talal Al-Samman, Ulrich Kerzel, <u>Sandra Korte-Kerzel</u> Institut für Metallkunde und Materialphysik, RWTH Aachen University

Our research journey commonly begins at the nanoscale, where we delve into the nanomechanics of hard phases within intermetallic crystals. By characterizing and modeling plasticity, we aim to unravel the underlying mechanisms that distinguish these materials from traditional metals. This exploration is crucial for identifying new descriptors of plasticity, which can guide us in selecting phases that achieve an optimal balance between high hardness and adequate toughness. Furthermore, this understanding paves the way for advanced defect engineering in functional intermetallics. Transitioning from the nanoscale to larger scales, our focus shifts to dual-phase microstructures composed of soft and hard phases. Here, larger datsets and machine learning techniques become instrumental in analyzing damage mechanisms during deformation processes. The inherent heterogeneity across various scales necessitates a combined approach that leverages both high-resolution imaging and large-area observations through electron microscopy and nanoindentation mapping to reveal phase properties. These methods not only enhance our data acquisition but also prompt a re-evaluation of how we interpret complex material behaviors based on microscopy and mechanical properties of individual phases from nanomechanical testing.

Abstracts - Oral Presentations

Day 3: Thursday, March 6th

Talks — Session VII

Invited Talk

09:30 Experimental assessment of the mechanical reliability of microelectronics using advanced micromechanical testing strategies

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The increasing complexity of loading conditions in modern microelectronic devices needs a deep understanding of their micromechanical behaviour. This study focuses on the experimental assessment of mechanical reliability in microelectronics, emphasizing the importance of identifying micro-mechanical failures. On one hand, we employ a combination of micromechanical testing with variable loading scenarios and acoustic emission techniques to investigate these critical aspects. As a representative case, we demonstrate the micro-mechanical loading of copper pillar structures, explaining our workflow. On the other hand, to enhance the interpretation of these results, we employ finite element modelling. This approach requires the parameterization of these FE models, which in turn relies on advanced material models that accurately represent the materials and interfaces involved. To derive these material models, we conduct advanced micromechanical experiments. For example, in-situ SEM nanoindentation experiments using a Bruker Hysitron PI87 system are utilized to extract small-scale interface properties. Furthermore, we will explore material behaviour at extreme temperatures, employing the Bruker Xsol stage to reach conditions as low as -120 °C, broadening the parameter space for our FE models. Additionally, we parameterize advanced material models for e.g. viscoplastic behaviour through inverse parameter identification techniques based on spherical nanoindentation experiments coupled with FEM. While the primary focus is on established materials, we also initiate research into the micro-mechanical behaviour of novel electronic materials, such as 2D polymers. This includes in-situ TEM experiments using a Bruker PI95, aimed to show fundamental mechanical properties of these emerging materials. Our findings will contribute to the development of more reliable microelectronic systems, ensuring their performance under complex operational conditions.red]

10:00 Measurement of interfacial toughness between Polycrystalline and Monocrystalline Silicon Carbide

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Electronics devices based on Silicon Carbide (SiC) are instrumental in the field of high voltage electronics for car electrification. The fabrication of monocrystalline SiC (monoSiC) ingots is renowned to be cost- and time-intensive, hence recent effort has been addressed to propose more sustainable and scalable alternative substrates. Among others, polycrystalline SiC (polySiC) substrates with a thin seed of monoSiC on top represent an attractive solution. On the other hand, as compared to a monolithic monoSiC substrate, the polySiC/monoSiC interface may act as an extra crack propagation path in fabrication steps inherently associated to severe mechanical stresses (e.g., wafer sawing for singulation of individual chips). In the view of risk assessment, it is worth to compare the adhesive toughness between polySiC and monoSiC against the cohesive toughness of the individual materials mating at the interface. In this study, the measurement of adhesion energy between polySiC and monoSiC has been pursued by nanoindentation.

A prismatic coupon was disk-sawn from a polySiC/monoSiC wafer. Focused ion beam milling was used to remove sawing damage and obtain smooth testing areas. Cross-sectional nanoindentation (CSN) proved effective to induce delamination, indicated by a displacement burst (pop-in) in the load/displacement signal. The interface toughness was estimated based on the load/displacement hysteresis and the extent of delaminated area, the latter assessed by means of ex-situ optical profilometry.

Following this procedure, polySiC/monoSiC adhesion energy was found to be comparable or higher than the cohesive toughness of monoSiC.

10:20 Study of epoxy-based molding compound degradation at high temperature operation using nano-indentation mapping techniques

<u>Falk Naumann</u>*, Marcel Mittag, Tino Stephan, Sandy Klengel Fraunhofer IMWS, Fraunhofer IMWS, Fraunhofer IMWS

Electronic devices are commonly encapsulated with epoxy-based molding compounds (EMC) for protection from external or environmental influences. It provides excellent electrical insulation and ensures the stable operation of electronic circuits. In addition, mechanical properties of the EMC's, including their adhesion strength and resistance to thermal cycling, are critical for the durability of the packaged devices. However, aging and oxidation processes of the epoxy are leading to changes of the material behavior when EMCs are exposed to high temperatures. This degradation can affect mechanical, thermal, or hydroscopic properties of the epoxy material and has influence on the reliability behavior of packaged electronic components. Therefore, in the current study two types of mold compound were locally analyzed by nano-indentation mapping of the mechanical properties in combination with fluorescence microscopy to determine potential local degradation mechanisms. For defined aging, various devices were thermally stressed at 175°C for 500-2000 hours or subjected to a thermal cycling test (3000 cycles at T=-50 /+150°C) and compared with pristine samples. In a first step, cross sections by focused ion beam techniques were prepared and the aging process was visualized through fluorescence optical microscopy, revealing affected areas at the EMC. Afterwards, the local mechanical material properties (indentation hardness and modulus) of the epoxy matrix and glass filler components were determined by indentation mapping at several locations. Statistical post processing was performed to separate the epoxy matrix and the silica particle properties by analyzing the statistical distribution from the mapping. In this case, shifts in the statistical distribution could be correlated with ageing effects. As a result, the study provides insights into the evolution and spread of material degradation which has significance for the selection of EMCs for electronic packaging.

10:40 Speeding up Micromechanics Sample Prep for Indentation Testing Using Ultrashort Laser Pulses

Georg Schusser, Falk Naumann, Richard Busch, <u>Thomas Höche*</u> Fraunhofer IMWS, Fraunhofer IMWS, Fraunhofer IMWS

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Laser micromachining is becoming increasingly attractive for various methods of microstructure characterisation as it is inherently free of elemental contaminations and has orders of magnitude higher ablation rates than ion beams. When ultrashort pulsed lasers are used, the heat affected zone can be limited to a little as a few 100 nm. A laser beam can be focused to a diameter of about 10 μ m and infeeds to within 2 microns are state of the art.

At Fraunhofer IMWS, for more than a decade, this theoretically seductive idea has been transferred into reality by developing microPREPTM (https://microprep.pro/), a dedicated Laser Class 1 tool for automated sample prep jointly with 3D-Micromac AG.

It will be showcased, that these methods can also be applied for the preparation of common test structures

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(compression pillars, cantilever or tensile structures) used for micro-mechanical testing.

For a selection of typical materials, several preparation advantages will be demonstrated:

First, laser machining is much faster than competing ion-beam based techniques, allowing for faster preparation of more samples and improving statistical significance of mechanical measurements.

Second, in a multi-stage sample prep workflow, laser machining can help making the use of (plasma) FIB tools much more efficient by speeding up the coarse machining prior to final ion beam shaping and polishing.

Third, when using a femtosecond laser, all inorganic materials can be processed, even when transparent at the wavelength of machining. Challenging materials such as sapphire, quartz glass, and SiC can be machined at high definition as well.

Fourth, as massive ablation requires reliable removal of debris, concepts for attaining samples as clean as possible (snow jet cleaning, sacrificial layers) are being used.

Fifth, using a dedicated, multi-axle motion system combined with a galvanometer scanner and a recipe-based GUI control software, 2,5 D objects can be reproducibly carved from a specimen.

Talks — Session VIII

Invited Talk

11:30 Spherical nanoindentation of nuclear steels

Anna Kareer

University of Oxford

Structural materials in the reactor core of nuclear reactors are subjected to irradiation damage from neutron flux, which significantly alters the mechanical properties of these materials. Understanding these changes is essential for assessing the long-term structural integrity of nuclear components. Nanoindentation offers a distinct advantage in nuclear applications, as it minimises the volume of radioactive material required for testing while allowing for the characterisation of mechanical properties in shallow ion-irradiated layers. Unlike Berkovich indentation, spherical nanoindentation enables the direct measurement of indentation stress-strain curves, providing valuable insights into post-yield behaviour and work hardening. Key post-yield characteristics, such as plastic instability, elevated work hardening rates and reduction in elongation, are critical for understanding the impact of irradiation on mechanical performance. This talk will explore the use of spherical nanoindentation on irradiated nuclear steels, emphasising its advantages in evaluating irradiation-induced changes in mechanical properties, particularly regarding post-yield plasticity mechanisms. The findings highlight the potential of spherical nanoindentation as an effective tool to capture irradiation induced mechanical property changes in both neutron and heavy-ion irradiated materials. The method may serve as an alternative to traditional uniaxial tensile testing especially in the context of using heavy-ion irradiation to replace long-term reactor campaigns.

12:00 Nanoindentation - why and when does the tip sharpness matter

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In recent years, nanoindentation has gained increased interest in measuring micro- to nano-scale features with different material properties. It is virtually impossible to measure the elastic modulus of e.g. the thin film without the substrate influence or micro-particles/fibers within composite materials. Ideally, both plastic and elastic deformation under the indenter should be localized within the measured feature and a key parameter changing the deformation field size is the tip radius. Presented research is aimed at quantifying the influence of the tip radius on the strain field under the indenter with use of numerical modelling of different tip geometries, whereas the tip sharpness, represented via the tip radius and angle, is present in the models. In addition,

three real experimental measurements with Berkovich indentation tips with different tip radii were performed on thin layer nanoindentation to validate the simulations and see the impact of tip sharpness/bluntness on the real measurement. The results confirm the existence of large elastically deformed zone, with a strong localization under the tip with a direct connection between the tip sharpness and deformation localization affecting the experiment. This effect shows that in cases of measurement of highly localized material properties, the sharp tip is highly advantageous while if the measurement is aimed on more homogenized, larger volumes of material, the blunt tip can be used, without wearing of fresh, new tips.

12:20 Registrierende Mikrohärteprüfung" in Halle (Saale) – memorizing the 'stone age' of nanoindentation

Matthias Petzold

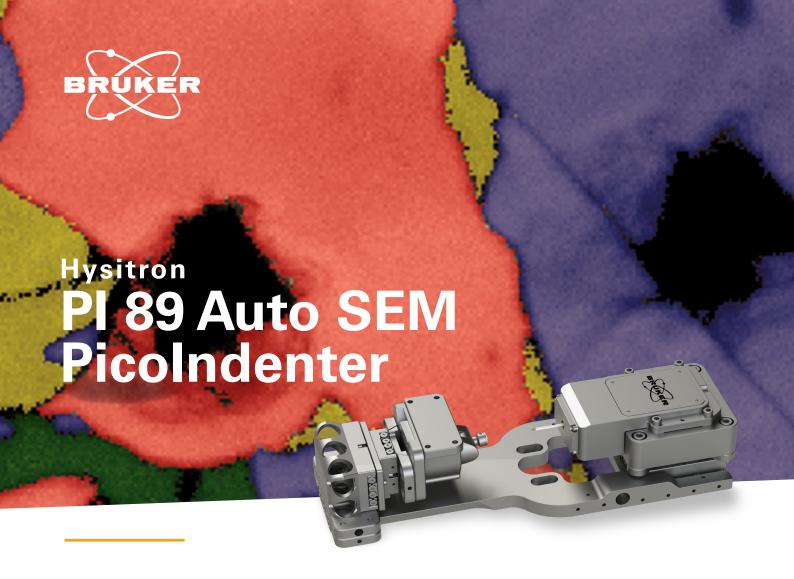
Fraunhofer Institute for Microstructure of Materials and Systems IMWS, Halle (Saale)

The method of nanoindentation with all its facets and variations has become indispensable in the world of materials science, especially for the investigation of small-scale high-tech materials. The "Nanobrücken 2025" meeting in Halle (Saale) will also provide an opportunity to look back at the beginnings of the technology, which had an important starting point right here in Halle.

The decisive metrological development step on the way to nanoindentation compared to conventional hardness testing was the introduction of a parallel and continuous measurement of indentation force and indenter depth. In particular, precise depth sensing made it possible to overcome the optical resolution limits of the light microscopic measurement of the remaining indentation size, which is a basic prerequisite for the characterization of thin films or measurements on small-area samples. Furthermore, the continuous recording of indentation curves also provided new additional information describing the complex deformation behavior of solids (e.g. elastic-plastic or time-dependent properties). As a result, quantitative material science parameters could be derived that support the mechanical modelling of materials also on a local scale, e.g. for thin layers.

In 1975, exactly 50 years ago, Fritz Fröhlich and Peter Grau, employees at the Department of Physics at Martin Luther University in Halle (Saale), were granted a GDR national patent for the first development of such a "Registrierende Mikrohärteprüfung" (or "recording" indentation testing). A first international scientific publication on the new technique followed in 1977.

From an anecdotal perspective of the author's own experiences in this research group, the presentation will report on the historical background to this invention at the time, the state of the technology used, early interpretations of the load-indentation depth curves and on the subsequent attempts to further commercialize the experimental setup as well as the associated limitations at that time under the given boundary conditions.

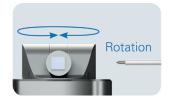


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Abstracts - Poster Presentations

Investigating the design of macromolecular-based inks for two-photon 3D laser printing

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Two-photon 3D laser printing (2PLP) is one of the most versatile methods for additive manufacturing of microto nano-scale objects with arbitrary geometries and fine features. With advancing technological capability and accessibility, the demand for new and versatile inks is increasing, with a trend toward printing functional or responsive structures. One approach for ink design is the use of a macromolecular ink consisting of a 'prepolymer' functionalized with photocrosslinkable groups to enable printability. However, so far the synthesis of pre-polymer inks for 2PLP often relies on an arbitrary choice rather than systematic design. Additionally, current structure–property relationship studies are limited to commercial or small molecule-based inks. Conventional formulations consist of a mixture of monomers, with no control of the resultant topology, resulting in limited tunability of the final mechanical properties. Herein, three macromolecular inks with varied compositions, molecular weights, and glass transition temperatures are synthesized and formulated into inks for 2PLP. 3D microstructures are fabricated and characterized in-depth with scanning electron microscopy as well as infrared spectroscopy and nanoindentation to enable the determination of structure–processability–property relationships. Overall, it is clearly demonstrated that the macromolecular design plays a role in the printability and mechanical properties of the obtained materials.

2. Nanomechanical studies on ZDDP based tibofilms grown on sapphire substrate

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To minimize wear in mechanical parts under relative motion, oils containing antiwear additives are commonly employed. Zinc-dialkyl-dithiophosphate (ZDDP), one of the most widely used antiwear additives, forms protective tribofilms that improve mechanical and tribological performance. Nanoindentation is a powerful technique for investigating the mechanical properties of these tribofilms.

In previous studies, nanomechanical properties of ZDDP tribofilms on bearing steel substrates were explored [1,2]. For the current study, polished sapphire substrates, which offer an exceptionally smooth surface, were investigated which allowing a deeper insight into the tribofilm's intrinsic properties. The formation of stable protective tribofilms is driven by temperature and tangential shear stresses at the contact interface. Tribofilms were grown using a tribometer setup, where a steel ball oscillated against a sapphire surface submerged in PAO 6 oil containing 5 weight % ZDDP. Tests were conducted in a heated bath at three temperatures: 30°C, 70°C, and 100°C. The test parameters included a track length of 2 mm, a steel ball with a 6 mm diameter, a load of 2 N, and a test duration of 15 minutes.

The tribofilms were characterized using microscopy and nanoindentation techniques. The low surface roughness of the sapphire substrate enabled precise indentation of the tribofilm, facilitating accurate nanomechanical measurements. Additionally, scratch tests using a conospherical tip under low loads were performed along the tribofilm to determine local friction values.

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3. Road to Failure: AFM indentation of polymers

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Indentation methods range from the macro to the nanoscale and with the decrease in scale more challenges arise. While researchers mostly understand micro- and nanoindentation methods, indenting with an atomic force microscope (AFM) is still relatively new with limited use in the chemistry or biology fields. However, AFM indentation has many uses in materials science and especially in polymer coatings. To illustrate the challenges of AFM indentation a case study on Epoxy-Silicon coatings with future aerospace applications will be shown. Compared to nanoindenters, AFM indentation has more crucial calibrations steps and specific data export routines for data analysis using contact mechanics models. AFM indentation has the advantage of utilizing mapping on an even smaller scale compared to modern nanoindenters and higher resolution surface imaging before and after. The AFM indentation results will be compared to Berkovich and Cube Corner indents made to similar depths and with similar loading-unloading functions. Differences in hardness and reduced elastic modulus between the measurement techniques as well as a proposed testing workflow will be presented.

4. Determining the elasticity of bacterial cells using microcompression tests

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Recently, there has been increased interest in the mechanical properties of eukaryotic and prokaryotic cells. While using nanoindentation for eukaryotic cells is quite common nowadays, this technique has not yet been well established for prokaryotic cells. One of the main challenges is the evaluation of the nanoindentation data for rod-shaped cells. The other challenge is their small size. In our study, we have addressed the gap in the evaluation by modifying Overbeck's mathematical model which is validated only for spherical cells [1]. This model was modified for the filled-torus geometry with the circular cross-section as the initial cell geometry. We have also optimized the imaging of the bacteria using fluorescent probe Bodipy 493/503 to better visualize each cell and be able to determine its sizes for the calculation of the elastic modulus (E). For the microcompression tests was used Hysitron BioSoft nanoindenter in displacement control mode equipped with the flat punch tips (Rc = $20~\mu m$) with the constant approach velocity $10~\mu m \cdot s - 1$. The fluorescence of the cells was visualized using inverted microscope Olympus IX73. For the measurements was used the producent of biodegradable polymers polyhydroxyalkanoates (PHA) Cupriavidus necator H16 together with its PHA non-producing mutant C. necator PHB-4. We immobilized both bacterial strains on the glass substrate,

fluorescently dyed and measured them in the liquid environment (phosphate buffer). Results indicate that the PHA producing bacteria (composed of 72% PHA) exhibit average E = 8,4 MPa which is 24% higher value than the non-producing mutant strain.

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5. Investigating the Effect of Nanoparticles on Toughness in Silicon Nitride Thin Films

<u>Filippo Sabatini</u>, Vincent Coutellier, Emanuele Cattarinuzzi, Paola Zuliani, Andrea Li Bassi Politecnico di Milano, STMicroelectronics

It is well established in the literature that increasing the silicon (Si) content in silicon nitride (Si-rich SiN) leads to the spontaneous formation of amorphous Si nanoparticles dispersed within the SiN matrix. While numerous studies have focused on the optical property

changes induced by these nanoparticles, little research has explored their impact on the mechanical properties. After initial characterization of three different SiN films with varying Si/N ratios, toughness measurements were pursued using nanoindentation: fracture toughness was estimated based on the length of indentation-induced cracks, comparing the length of the cracks propagated on a SiN step from an indentation on the partially uncovered substrate (Xia et al, 2004 – Acta Mater). The latter proved to be a valid method for measuring the toughness of thin films, giving new insight on the dependency of the SiN mechanical behavior at varying silicon content.

6. Viscoelastic Performance of Cellulose Nanofiber-Reinforced Bio-Nanofilms Under Extreme Conditions

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This study investigates the high-temperature viscoelastic properties of cellulose nanofiber (CNF)-reinforced nanofilms, which are critical for understanding their performance under extreme environmental conditions. The viscoelastic behavior of the bio-nanofilms was analyzed using a dynamic mechanical thermal analyzer (DMTA) in tension mode. The bio-nanofilms were prepared by blending polyvinyl alcohol (PVA) and chitosan (CS), reinforced with varying amounts of cellulose nanofiber (0, 3, 5, 7, and 8 wt.% relative to the PVA/CS weight) through the solvent casting method. The effect of CNF reinforcement on the films' storage and loss moduli was thoroughly evaluated.

The results show that the storage modulus of the bio-nanofilms significantly increased with CNF reinforcement compared to the control PVA/CS films. For instance, the storage modulus of the control group at 100°C was 182.3 MPa, while 7 wt.% CNF reinforcement increased it to 860 MPa. A similar trend was observed in the loss modulus. At 88.1°C, the control group had a storage modulus of 34.1 MPa, whereas the 5 wt.% CNF-reinforced films exhibited a maximum storage modulus of 411 MPa. At 225°C, the storage modulus of the control group was 34.1 MPa, while 7 wt.% CNF reinforcement increased it to 69.9 MPa. However, at 8 wt.% CNF reinforcement, both the storage and loss moduli decreased due to CNF agglomeration.

Overall, the CNF reinforcement significantly enhanced the viscoelastic behavior of the bio-nanofilms, demonstrating their potential for applications requiring high-temperature stability. These findings contribute to advancing material characterization techniques, aligning with the conference's focus on mechanical testing and material performance under extreme conditions.

7. Determining the influence of cooling 1.7225 on the microstructural properties using nanoindentation

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Dynamically high stressed components are often produced by forging and are characterised by a high service life due to the fine-grained microstructure. Premature failure of forged components is often due to residual stresses, which are usually caused by uncontrolled cooling after the forging process. A numerical analysis of the heating, forging and cooling process chain to predict the microstructure and the associated residual stresses can only be solved with a high computational effort. For this reason, a multi-scale simulation model is aimed at, with which the residual stresses are calculated on the basis of the change in the microstructure. This requires mechanical parameters as well as the distribution of the microstructure. To determine the required information, specimens were produced with different heat treatments. The resulting microstructure was examined and analysed using XPM mappings and metallographic analyses. Figure 1 shows an example of the influence of cooling in the edge zone of the specimen. The results were subsequently used for multi-scale residual stress simulations.

8. Quantitative characterisation of the mechanical and electrical properties of nanowires used for nano-energy harvesting

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Nanowire-based energy harvesting presents a promising solution for generating small-scale power in remote applications. However, the characterization of semiconductor nanowires (NWs) with high aspect ratios and sub-micron diameters requires precise quantification of their mechanical and electrical properties. This study utilizes the nanoindentation instrument (Hysitron Triboindenter TI-950) with electrical contact resistance (ECR) measurement capability to investigate the nanomechanical and nanoelectrical behavior of vertically aligned NWs. The instrument, incorporating a capacitive force transducer, conductive diamond indenter, and a Keithley SourceMeter, was rigorously calibrated to ensure traceability in force and electrical measurements. The results validate the feasibility of conductive nanoindentation for quantitative nanoelectromechanical characterization, contributing to advancements in NW fabrication and model validation. Future work aims to enhance measurement sensitivity and characterize ZnO nanowires for piezoelectric energy harvesting, further advancing nanometrology and sustainable energy applications.

From Atoms to Applications: Unraveling Coating Failure through Multi-Scale
 Analytical Modeling combining Molecular Dynamics, Stress Evaluation and Surface Experiments

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A better understanding of your coating stacks and their behavior in the application is crucial for the optimization of complex engineering systems. In this context, a modeling approach, in conjunction with targeted laboratory and functional tests, is particularly attractive as it can accelerate the coating selection and achieve its goals within the desired application field. Indeed, this methodology helps to understand the coating

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system, all its coating layers and interfaces including the substrate. Simulations eludicate coating limitations and guide the refinement of the coating architecture within a defined framework.

This presentation will showcase how the required data can be obtained by analyzing mechanical surface tests such as tribo-wear, indentation and scratch measurements. The analytical models developed by SIO dramatically speed up the simulation and optimization of complex contact conditions with less computing power. An advantage being that analytical models are bot invertible and scale invariant, with application to simulations in a great variety of fields from: tectonic plates, large area coatings, engine parts, turbine blades, hard-disks and even the orbital layers of 2D materials. Graphene [1] provides an example of the latter, where the opportunity to visualize stress and strain from charged ions to dopants provides opportunity for reverse optimization.

In the second segment, we will explore our ongoing project in a different field. The work on 2D materials pushed us toward small scale effects, leading us to dive into molecular dynamics simulations. The calculation of interactions between atoms and molecules and understanding how these influence failure will bring new insights to understand the macroscopic material behaviour in both experimental and applied fields.

[1] https://siomec.com/applications/graphene

10. Advanced feedback modes in nanomechanical testing

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Requirements for faster and more intelligent feedback functions are driven by the need for higher resolution and control of strain rates. Three examples of variations in advanced control modes are given: two increasing nanomechanical mapping performance and one increasing the accuracy of creep and/or frequency sweep data in thin films. Feedback triggers, switching of feedback modes (force and displacement), and the use of stiffness as a feedback parameter are presented.

11. In-Situ SEM Push-To-Pull Testing of a Graphene Sheet

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Key Words: Graphene, Push-to-pull, Raman spectroscopy, Scanning electron microscopy, Molecular dynamics

Graphene, known for its exceptional physical properties, exhibits remarkable mechanical characteristics, including a theoretical elastic modulus of 1TPa. In-situ scanning electron microscopy nanomechanical testing utilizing push-to-pull (PTP) devices has enabled scientists to explore the elastic and fracture properties of graphene monolayers. In this study, we focus on the experimental aspects of PTP tensile testing, particularly highlighting the none-uniform strain field observed over the width of graphene samples. Utilizing spatially resolved Raman-derived strains corrects for the strain none-uniformity and helps aligning experimentally measured elastic properties of monolayer graphene with prior experimental and theoretical estimates of the elastic modulus. In our molecular dynamic simulations (MD), we investigate the effects of strain heterogeneity and varying adhesion levels between graphene and silicon oxide substrates on stiffness measurements during gradual straining. Our simulations incorporate uneven adhesion distributions and lattice defects (grain boundaries vs. vacancies) to analyze stress-strain curves and identify atomic-level stress concentrators contributing to premature graphene failure under PTP conditions.

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12. Insights into Dispersed Mechanical Properties in Complex Steel Alloys via Nanoindentation Property Mapping

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Accelerated property mapping by nanoindentation testing involves performing a large number of fast indentation tests at the nanoscale, with spacings ranging from 50 nm to several micrometers, at a rate of more than one indentation per second. Combining the locally measured properties, such as hardness, reduced modulus, creep, and many more, provides direct access to the dispersed mechanical properties with high spatial resolution.

A Hysitron TI980 Tribolndenter was used for the indentation experiments. Three case studies on steel samples are presented, highlighting new insights into the interplay between the material's nanoscale mechanical properties and its mechanical behavior from the micro to macroscale. The studies include a comparison of nanoindentation hardness mapping and micro-Vickers hardness tests, a comparison of nano-hardness mappings of complex phase steel alloys and their deformability, and a machine learning approach to identify clusters of similar behavior in a complex alloy.

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13. Study of thermomechanical stability of plasma deposited thin films using in situ high temperature nanoindentation

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This study investigates the thermally induced changes in the mechanical properties of Si-, O-, and Cu-doped diamond-like carbon and plasma polymer thin films, as well as their relationship with changes in structure, chemical composition, and physical properties. The mechanical properties of the selected materials were evaluated using a Hysitron TI 950 instrument equipped with an xSoI 800 stage (Bruker). Hardness and reduced elastic modulus were measured at both room temperature and elevated temperatures. Thermal dependence of the mechanical properties was assessed in 50°C increments, starting from 24°C up to 600°C. At each temperature step, system calibration procedures were performed after every 10 indentations. This research has been supported by the Czech Science Foundation under project GA23-06263S. We acknowledge CzechNanoLab Research Infrastructure supported by MEYS CR (LM2018110).

14. Doping strategies to enhance micromechanical strength in sol-gel-derived metal oxide semiconductors

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In applications where metal oxide semiconductors are in contact, their mechanical properties play a pivotal role. This study addresses the need for enhanced mechanical performance, which is crucial for practical applications, and proposes a comprehensive optimization strategy. The investigation focuses on three widely used semiconductors: zinc oxide (ZnO), titanium dioxide (TiO₂), and tin dioxide (SnO₂). All metal oxides were synthesized using the conventional sol-gel method, recognized for its cost-effectiveness, simplicity, rapid processing, and environmentally friendly nature compared to other techniques. Despite their robust nanomechanical characteristics, sol-gel-derived metal oxides often exhibit poor micromechanical properties due to their granular microstructure, which contains pores and voids between grain clusters. To overcome these limitations, various strategies, such as heat treatment, pressure-assisted processing, and doping with different elements or compounds have been developed to improve their mechanical performance. This study specifically investigates the impact of cobalt and copper co-doping on the structural and mechanical properties of these metal oxides. A range of characterization techniques, including X-ray diffraction, scanning electron microscopy, energy-dispersive spectroscopy, Brunauer-Emmett-Teller analysis, and Vickers indentation, was employed to evaluate the effects of co-doping. The analysis of the synthesized samples demonstrates significant improvements in both structural and mechanical properties. The co-doping process, applied at a 3% Co-Cu doping ratio, effectively enhances the hardness and elastic modulus of each metal oxide by inducing structural modifications, such as reduced porosity and grain size refinement. Beyond identifying optimal processing parameters for each metal oxide, this study also presents a comparative analysis, addressing a gap in the literature and providing valuable insights into the field.

Keywords: Sol-Gel-Derived Metal Oxides, Transition Metal Doping, Vickers Indentation

15. Surface Investigation by Atomic Force Microscopy of a Li-ion battery electrode

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The energy storage system is recognized as an increasingly important parameter in electricity and energy systems¹. The increased demand for electricity and energy has led to the development of efficient storage systems. This is why battery technologies have improved significantly to meet practical electric vehicles' challenges and circumvent the intermittency and variability of renewable energy sources. During the last decades, battery technologies have improved considerably to meet practical electric vehicles' challenges and circumvent the intermittent variability of renewable energy sources.

Lithium-ion batteries (LIBs) are currently considered the most suitable energy storage device for powering electronic devices owing to their attractive properties, including high energy efficiency, lack of memory effect, long cycle life, high energy density, and high power density². These advantages allow them to be smaller and lighter than conventional rechargeable batteries. During the initial stages of LiB operation, the so-called Solid Electrolyte Interphases (SEI) start to grow at the electrodes' surface. It corresponds to an interfacial layer resulting from the electrochemical reduction of the solvent, salts, and impurities. This SEI layer plays a crucial role in the long-term performance of LIBs. Thus, SEI formation and electrochemical stability over long-term operation should be a primary topic of future investigation in LIB development³.

Atomic Force Microscopy (AFM) is considered one of the most demanding techniques to get maximum

information about the various surface properties of the LiB electrodes, such as topography, mechanical, electrical, electrochemical, and surface potential⁴. To follow up the different properties, AFM delivers different modes such as Quantitative Nanomechanical Microscopy, Kelvin Probe Force Microscopy (surface potential), Scanning Electrochemical Microscopy (electrochemistry), and TUNA conductivity (electrical properties). Using AFM and its different modes allows a depth analysis of the surface properties and interfaces of the electrode. To understand deeply, we need to study all the parameters in this study⁶.

From the figure we can see the obtained mechanical properties of the one of the most used electrode material that is HOPG (Highly Oriented Pyrolytic Graphite) with a scan rate of 0.5 Hz inducing the information about the DMT module and the adhesion.

Nowadays, active materials such as LiNiMnCoO₂ (NMC) are frequently used in the batteries field, which has become an essential topic for understanding the surfaces of these active materials⁷. The results show that the scan rate plays a crucial role in decreasing the artifacts that occur during the mechanical properties of the active electrodes⁸. Playing different scan rates and the velocity of the tips can improve the various constraints involved in the mechanical measurement, such as DMT module, Indentation, and adhesion. The results also conclude that it's very important to make compromises between the applied parameters. This study will contribute to the surfaces information during the cycling of the active material electrode to determine and study the interfaces, which are considered as a very important part of the battery world.

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- 16. Stability of nanoparticles with a focus on their morphology, and mechanical properties with the dynamics of proteins as well as other biomolecules for health diseases

<u>Seniha Simale Su Uygan</u> HZDR

Atomic force microscopy is a unique tool for molecular imaging in the field of nanotechnology, biotechnology, and nanobiotechnology. In particular, nanoparticles have been widely used in various applications such as biomedicine, electronics, environmental remediation, biosensors, and gas sensors. Moreover, protein molecules have been visualized during their functional activity at high spatiotemporal resolution. Thus, mechanistic insights for molecular processes of proteins with a focus on nanoparticle interactions have been investigated through the combination of atomic force microscopy, and molecular dynamics with a focus on software development, and experimental skills. Function, variability, and assembly of proteins throughout their interactions with nanoparticles have been observed different morphology, stability, and dynamics with a focus on mechanical properties. In particular, the interaction forces of silica

nanoparticles as electrostatic repulsion forces, van der Waals attractive forces, and steric repulsion forces are related on the stabilization mechanisms of nanoparticles such as carbon nanotubes, gold, and silica nanoparticles. Moreover, it plays a crucial role in order to quantify their interaction forces with proteins in addition to their mechanical properties. Therefore, nanoindentation is the most important process to perform mechanical testing for these nanoparticle-protein-biomolecule structures. Furthermore, it greatly extend the range of experiments possible for probing biological systems at the molecular level. For instance, lateral resolution of 0.5–1 nm and vertical resolution of 0.1–0.2 nm have been observed. Overall, it observes various challenges to explore the most effective hybrid structures in terms of human health for real-world applications.

17. Predicting the microstructural evolution of ion-irradiated Eurofer97: Nanoindentation study supported by CPFEM and TEM

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Reduced activation ferritic/martensitic (RAFM) steels are the main candidates for the construction of structural components in future fusion and Gen IV nuclear reactors. To ensure safe and stable reactor employment, RAFM-based materials require efficient methods for their characterization under harsh operational conditions. Constantly enduring neutron irradiation, their mechanical properties degrade and may cause a failure of the component. However, neutron irradiation for research purposes is an expensive and long process, so it becomes a major limiting factor to steadily investigate its effect and deliver new research data. Hence, a safer, more paced, and cheaper solution of ion irradiation as a tool for surrogating the neutron damage is becoming more and more popular. As ions are characterized by their penetrating ability, the introduced damage is non-uniformly distributed and densely accumulated on the sub-surface. To correctly estimate their impact, nanoindentation technique is widely applied. The presented study demonstrates a semi- empirical approach to effectively interconnect the ion and neutron radiation-induced hardening in RAFM steels, introduced in a range of irradiation conditions. The applied set of tools, based on nanoindentation and tensile tests, as well as their simulations using crystal plasticity finite element method, allows us to extract the irradiation effect on the material law, and accurately reproduce the experimental data. Ultimately, the analysis performed on an ion-irradiated specimen can provide the macroscale (neutron irradiated) yield stress values in a range of dpa doses. The complementary investigations of the microstructural evolution done by focused ion milling and transmission electron microscopy are compared with their computational analogue to confirm the predictive capability of the method.

Practical Information

Nanobrücken 2025

Nanobrücken 2025 will be held at the Fraunhofer Institute for Microstructure of Materials and Systems IMWS, located in the beautiful city of Halle.

Nanobrücken, Bruker's annual Nanomechanical Testing Conference and User Meeting for international researchers and industrial leaders in nanoindentation and nanotribological testing, includes oral presentations from leading research groups, as well as live demonstrations and discussions with Bruker experts.

For additional information, please visit the conference website at www.bruker.com/Nanobruecken.

Oral Presentation Guidelines

The workshop prefers that you use our conference computer. Please bring your presentation file on a memory stick. The presentation screen aspect ratio is 16:9.

Student talks are 12 minutes in length, with an additional 3 minutes for discussion.

Contributed talks are 15 minutes in length, with an additional 5 minutes for discussion.

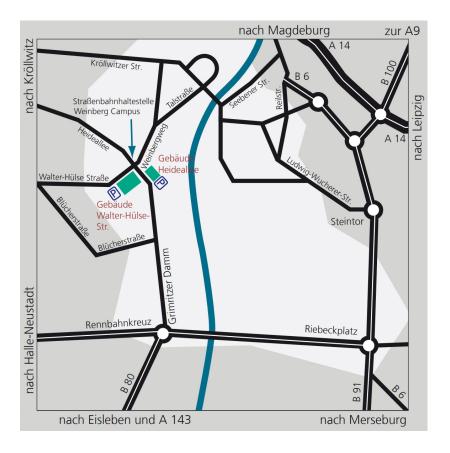
Invited talks are 25 minutes, with an additional 5 minutes for discussion.

Poster Presentation Guidelines

Posters should be prepared in A0 portrait format; stands and pins will be provided.

Conference Venue

Fraunhofer Institute for Microstructure of Materials and Systems IMWS Walter-Hülse-Straße 1 06120 Halle (Saale), Germany





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