BRUKER

Nanobrücken 2023

A Nanomechanical Testing Conference and Bruker User Meeting

May 23rd - 25th, 2023 | Saarbrücken, Germany

Nanobrücken 2023 Program

Preface

Saarland University and Bruker are pleased to announce that **Nanobrücken 2023** will take place May 23-25 in the beautiful city of Saarbrücken.

This is the 13th 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

Saarland University
Aula der UdS (Auditorium of the US)
Campus A3 3
66123 Saarbrücken, Germany

Program Committee

Conference Committee Members

Prof. Christian Motz, Saarland University (Germany)
Dr. Florian Schaefer, Saarland University Germany)
Dr. Jaroslav Lukeš, Bruker (Czech Republic)
Dr. Ude Hangen, Bruker (Germany)

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Bruker Organizational Contacts

Dr. Rhys Jones, Bruker (United Kingdom)
Annette Niedballa, Bruker (Germany)

Day 1: Tuesday, May 23rd

Welcome

10:00 Opening Remarks
Jaroslav Lukeš and Ude Hangen, Bruker

Talks — Session I

Chair: Florian Schaefer, Saarland University

Invited Talk

10:15 "Accounting for Thermal Drift During Cyclic Nanoindentation: a Methodological Approach for Local High Cycle Fatigue Investigations on Metals and Ceramics"
Claudia Fleck, Technische Universität Berlin

10:45 "High-throughput fracture testing: A novel strategy enabled by direct laser writing and nanoindentation mapping"
Alexander Jelinek, Montanuniversität Leoben

11:00 "Micromechanical characterization of tantalates and niobium oxide observed in refractory metal-alumina composite materials"
Gökhan Günav, TU Bergakademie Freiberg

11:15 "Nanoindentation study of the oxide scale on FeCr alloy by high-pressure torsion"
Kuan Ding, Technical University of Darmstadt

11:30 "Characterization of wear particle generation by in-situ SEM micro-scratching of mono-crystalline silicon"
Tom Bertens, Eindhoven University of Technology

11:45 "Micromechanical in-situ nanoXCT studies to examine fracture failure mechanisms of on-chip interconnect stacks"
Stefan Weitz, Fraunhofer IKTS

12:00 "Bruker Nanoindentation Instruments"
Sanjit Bhowmick, Bruker

Lunch Break

12:20 Lunch Provided On-Site

Talks — Session II

Chair: Holm Geisler, GlobalFoundries Dresden Module One LLC & Co. KG

13:30 "Surface integrity on ground WC-Co hard metal grades under service-like working conditions"
JJ Roa, Steros GPA Innovative S.L.

13:50 "Fracture toughness of cementitious composites assessed via nano-scratch"
Jiří Němeček, Czech Technical University in Prague

14:10 "Examining local fracture toughness of grain size tailored WCu nano-composites"
Klemens Schmuck, Montanuniversität Leoben

14:25 "Micro- and meso-mechanical fatigue testing – abnormal grain growth, crack initiation and growth"
Jutta Luksch, Saarland University

14:40 "Measuring interface adhesion through nanoindentation and nanoscratching"
Satha Almarri, University of Oxford

14:55 “Tracking the evolution of local elastic strain fields in tailored metallic glass composites during in-situ deformation in the TEM”
Simon Felnner, Austrian Academy of Sciences

15:10 Short Break / Coffee Provided

Talks — Session III

Chair: Mathilde Laurent-Brocq, Institut de Chimie et des Matériaux Paris Est, UPEC-CNRS

15:40 “Mechanical property changes accross the spin transition in a molecular film probed by nanoDMA”
Maryam Naximsobban, CNRS and University of Toulouse

Invited Talk

15:55 “Novel approaches to avoid FIB-artifacts in microcantilever fracture tests”
Subin Lee, Karlsruhe Institute of Technology

16:25 “Mapping strain fields in CuZr-based metallic glasses by 4D-STEM assisted in-situ mechanical testing”
Lukas Schretter, Austrian Academy of Sciences

16:40 “Initial Analysis on the Statistics of Serrations and Strain-Rate Sensitivity of Vit105”
Valeria Lemkova, University of Saarland

Poster Session and Barbecue

All Posters are Eligible for Top Poster Prize

16:55 Poster Session (All posters, see list on page 6) / Barbecue

Keynote Lecture

Chair: Christian Motz, Saarland University

19:00 **“Probing defect - defect interactions in metals and (complex) alloys by in-situ nanomechanics”**
Prof. Dr. Gerhard Dehm, MPI für Eisenforschung and Ruhr University Bochum

Day 2: Wednesday, May 24th

Talks — Session IV

Chair: Claudia Fleck, Technische Universität Berlin

Invited Talk

09:30 “Nanoprobing water inclusive materials”
Karlis Gross, Riga Technical University

10:00 “Humidity-Dependent Mechanical Properties of Halide Perovskites”
Sidney Cohen, Weizmann Institute of Science

10:20 “Evolution of Mechanical Strains in Biological Ceramics”
Shahrouz Amini, Max Planck Institute of Colloids and Interfaces

10:40 “Exploring the micromechanics of the strongest known biomaterial: How the limpet tooth realizes a higher strength than hardness”
Michael Wurmshuber, Montanuniversität Leoben

11:00 “Compression of pellets from filamentous microorganisms”
Achim Overbeck, Technische Universität Braunschweig

11:20 Short Break / Coffee Provided

Talks — Session V

Chair: Subin Lee, Karlsruhe Institute of Technology

Invited Talk

11:50 "Nanoindentation on Microchips: Thermomechanical Characterization beyond Hardness and Reduced Modulus"
Holm Geisler, GlobalFoundries Dresden Module One LLC & Co. KG

12:20 "Characterization of nanomaterials using On-Axis TKD in SEM: challenges and benefits"
Daniel Goran, Bruker Nano GmbH

Invited Talk

12:40 "Mechanical Properties of 2D-like Polydopamine Films"
Emerson Coy, Adam Mickiewicz University

Lunch Break

13:10 Lunch Provided On-Site

Talks — Session VI

Chair: Gerhard Dehm, MPI für Eisenforschung and Ruhr University Bochum

Invited Talk

14:30 "Nanoindentation, a powerful tool to study strengthening in high entropy alloys"
Mathilde Laurent-Brocq, Institut de Chimie et des Matériaux Paris Est, UPEC-CNRS

15:00 "Development of novel nanoindentation-based stress relaxation tests to study transient plasticity in metals"
Gaurav Mohanty, Tampere University

15:20 "Revealing deformation mechanisms by scale-bridging mechanical testing from in situ electrochemical nanoindentation to strain rate jump tensile tests"
Florian Schaefer, Saarland University

15:40 "Detecting local hydrogen content in materials by scanning Kelvin probe force microscopy"
Christian Motz, Saarland University

16:00 Short Break / Coffee Provided

Talks — Session VII

Chair: Oden Warren, Bruker

16:30 "Multi-scale mechanical testing and high-resolution microstructural characterization: Investigation of deformation mechanics of thin hard coating"
Idriss El Azhari, Saarland University and Universitat Politècnica de Catalunya

16:50 "Nanomechanical investigations of Tribofilms formed by ZDDP and Phosphonium-based ionic liquids on Bearing surfaces"
Florian Pape, Leibniz University Hanover

17:10 "Adaption of TI 950 for microfretting experiments"
Dominic Linsler, Fraunhofer IWM MikrotroboligeCentrum µTC

17:30 End of Conference

19:30 Conference Banquet Dinner

Day 3: Thursday, May 25th

Practical Sessions

09:30	Opening – Group Assignments
09:45	"Tribo iQ—Visualize Your Results" Ude Hangen, Bruker
10:15	Short Break / Coffee Provided
10:30	Hands-on Demonstration and Applications Discussion Hysitron TI 980 TriboIndenter with David Vodnick, Bruker Hysitron PI 89 SEM Picoindenter with Sanjit Bhowmick, Bruker
12:30	Farewell snack: Saying goodbye with a delicious treat

Poster List — Ordered by last name of presenting author

1. "Calo Indent and Droplet Erosion Module – New methods to evaluate stress distributions and profiles"
   N. Blerwisch, Saxonian Institute of Surface Mechanics
2. "PeakForce QNM, the pathway to interrogate nanomaterials"
   Isaac Brotons-Alcázar, Universitat de València
3. "Investigations of the joining zone on hybrid profiles"
   N. Heimes, Leibniz University Hannover
4. "Frictional properties of oxide scale under hot forming conditions on a nano level"
   J. N. Hunze, Leibniz University Hannover
5. "Experimental tips for reliable measurement of mechanical properties on hard coatings by nanoindentation"
   T. de Souza Lamim, Luxembourg Institute of Science and Technology
6. "Rose Prickles: Multiscale Structure-Mechanics-Function Relationships"
   Liat Levavi, Ben-Gurion University of the Negev
7. "Nanomechanical measurement of near-surface and interfacial properties for polymeric additive manufacturing materials"
   Zhi Li, Physikalisch-Technische Bundesanstalt
8. "The nanoindentation measurements influenced by surface topography"
   Tomas Plichta, Czech Academy of Sciences
9. "Performance evolution of inorganic fullerene nanoparticle lubrication additive using PI 95 in-situ TEM nanointender"
   Pattamadai Sundaram Sankara RK, INSA Lyon

Abstracts - Oral Presentations

Day 1: Tuesday, May 23rd

Talks — Session I

Chair: Florian Schaefer, Saarland University

Invited Talk

10:15 "Accounting for Thermal Drift During Cyclic Nanoindentation: a Methodological Approach for Local High Cycle Fatigue Investigations on Metals and Ceramics"

Claudia Fleck[1,*], Merle Schmahl[1], Cecilia Müller[1], Reinhard Meinke[1], Erika G. Alves Alcantara[1], Ude Hangen[2]

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Local microstructural inhomogeneities strongly affect the fatigue behaviour of materials. Cyclic nanoindentation allows the characterisation of the influence of single phases and their interactions on fatigue mechanisms on a local scale. Here we report on a method for high cycle fatigue testing by nanoindentation. The combination of high- and low-frequency indentation modes enables us to reach high cycle numbers and to obtain high enough numbers of data points to reconstruct force-displacement hysteresis loops. During the relatively long nanofatigue tests, the stochastic course of thermal drift is a challenge. To overcome the corresponding artefacts in the displacement measurement, we measure the drift rate in low-force holding segments that are interspersed in regular intervals. With these drift rates we then correct the displacement values. Thus, we achieve reproducible measurements of the cyclic deformation data. This we show for two very different materials, a ductile metal and a brittle ceramic.

10:45 "High-throughput fracture testing: A novel strategy enabled by direct laser writing and nanoindentation mapping"

Alexander Jelinek[1,*], Stanislav Zak[2], Daniel Kiener[1], Megan J. Cordill[2], Markus Alfreider[1]

1. Department Materials Science, Chair of Materials Physics, Montanuniversität Leoben, Franz-Josef-Straße 18, Leoben 8700, Austria
2. Austrian Academy of Sciences, Erich Schmid Institute for Materials Science, Jahnstraße 12, 8700 Leoben, Austria

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Two-photon lithography (TPL) enables the fabrication of micrometre sized objects with submicron precision. Using modern printing devices, this process can be automated to a large extend. The same holds true for the mechanical testing employing a common nanoindentation setup. Combining these two technologies with a suitable data evaluation procedure, for the first time the testing of a vast number of specimens becomes feasible with comparably less effort. The mechanical loading scenario we choose is using a push-to-pull (PTP) geometry, incorporating a double edge notched tension specimen geometry in mode I, to determine the essential work of fracture (EWF) of the constituent photoresist. Within the presented work, multiple specimen grids, including initially 2100 individual PTP specimens, were fabricated and compression tested via a flat punch indenter tip using a Hysitron TS 77 system to investigate processing influences on the resulting fracture properties. The compensation for the PTP frame stiffness and EWF evaluation of whole specimen grids were performed by a python script to obtain statistically representative results. Based on the presented methodology, the authors aim to establish a framework for statistical fracture evaluation of photoresist materials, and open further possibilities for characterization of similarly processed materials as well as applied thin film coatings.

11:00 "Micromechanical characterization of tantalates and niobium oxide observed in refractory metal-alumina composite materials"

Gökhan Günay[1,*], Robert Lehnert[1], Michael K. Eusterholz[2,3], Torben Boll[2,3], Horst Biermann[1], Anja Weidner[1]

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2. Institute for Applied Materials – Materials Science and Engineering (IAM-WK), Karlsruhe Institute of Technology (KIT), Engelbert-Arnold-Str. 4, 76131 Karlsruhe, Germany
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In this study, refractory metal composites based on alumina and refractory metals such as tantalum and niobium were studied. These materials were manufactured via casting followed by pressureless sintering at 1600°C for 4 h [1]. During the sintering process, additional phases such as tantalates ($AlTaO_4$) and niobium oxides (NbO) were formed in the microstructure [2]. Mechanical properties of these composites were studied at high temperatures (up to 1500°C) under compressive loading [3, 4]. Since these phases may have a significant effect on mechanical properties of the bulk material, it is necessary to understand their mechanical behavior. To the best knowledge of the authors, no detailed information on mechanical properties of tantalates and NbO can be found in the literature, so far. Nanoindentation is an effective method to characterize the micromechanical behavior of these phases - $AlTaO_4$ and NbO - at room temperature. In the scope of this study, several nanoindentation measurements were carried out both on Ta, Nb and Al_2O_3 grains as well as on AlTaO4 and NbO phases. In addition, scanning electron microscopy observations (electron backscattered diffraction) were performed on selected regions after indentation. The results show that Al_2O_3 has, as expected, the highest indentation hardness. In contrast, the pure refractory metals tantalum and niobium are characterized by the lowest values. However, whereas the hardness values of the tantalates is in between Ta and Al_2O_3, the indentation hardness of NbO is very close to that of Al_2O_3. Both NbO and Al_2O_3 behave brittle, which is well documented by many cracks in both phases in initial state before any deformation. Thus, the brittle NbO seems to be more detrimental for mechanical properties compared to $AlTaO_4$.

[1] T. Zienert, D. Endler, J. Hubálková, P. Gehre, M. Eusterholz, T. Boll, M. Heilmaier, G. Günay, A. Weidner, H. Biermann, B. Kraft, S. Wagner, C. G. Aneziris, "Coarse-Grained Refractory Composite Castables Based on Alumina and Niobium," *Advanced Engineering Materials* 24 (8), Special Section "Refractory Composites" (2022).

[2] M. K. Eusterholz, T. Boll, J. Gebauer, A. Weidner, A. Kauffmann, P. Franke, H. J. Seifert, H. Biermann, C. G. Aneziris, M. Heilmaier, "High-Temperature Ternary Oxide Phases in Tantalum/Niobium–Alumina Composite Materials," *Advanced Engineering Materials* 24 (8), Special Section "Refractory Composites" (2022).

[3] A. Weidner, Y. Ranglack-Klemm, T. Zienert, C. G. Aneziris, H. Biermann, "Mechanical High-Temperature Properties and Damage Behavior of Coarse-Grained Alumina Refractory Metal Composites," *Materials* 12, 3927 (2019).

[4] G. Günay, T. Zienert, D. Endler, C. G. Aneziris, H. Biermann, A. Weidner, "High-temperature compressive behavior of refractory alumina-niobium composite material," *Advanced Engineering Materials* 24 (8), Special Section "Refractory Composites" (2022).

11:15 "Nanoindentation study of the oxide scale on FeCr alloy by high-pressure torsion"

Kuan Ding[1,*], Enrico Bruder[1], Christian Dietz[1], Karsten Durst[1], Xufei Fang[1]

1. Department of Materials and Earth Sciences, Technical University of Darmstadt, 64287 Darmstadt, Germany

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High-pressure torsion (HPT) processed Fe-15wt.%Cr alloy with ultrafine-grained microstructure (UFG) was used to study the oxidation resistance and mechanical properties of the oxide scale. The microstructure before and after the HPT process was characterized using Electron Backscatter Diffraction (EBSD), and the hardness was measured using nanoindentation. After oxidation at 600°C for 15 h, thin and dense oxide scales formed on top of both coarse-grained and UFG FeCr samples. The microstructure and composition of the oxide scale on top of the substrate with and without the HPT process were investigated using Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM), and Energy-Dispersive X-ray Spectroscopy (EDS). Furthermore, the formed oxide scale was characterized using nanoindentation pop-in study, which suggested dislocation nucleation, crack formation, and oxide scale penetration events occurred during indentation. The results show that the oxide scales have excellent protection for the metallic substrate, and the HPT process improves the oxidation resistance. Our work proves that HPT processing can improve both the mechanical properties and the high-temperature corrosion resistance for potential high-temperature applications.

11:30 "Characterization of wear particle generation by in-situ SEM micro-scratching of mono-crystalline silicon"

T. Bertens[1,*], S.O. Sperling[1], K. van den Broek[2], M.G.D. Geers[1], J.P.M. Hoefnagels[1]

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According to Moore's law, the miniaturization of Integrated Circuits (ICs) is an ongoing process where semiconductor manufactures strive for nano-scale resolution using innovative fabrication techniques. In this pursuit, a clear understanding of contamination sources is essential since it governs the fabrication yield and reliance of ICs. In recent years, contamination sources at contacting machine components are found and the occurring wear particle generation is investigated more closely. The interaction between silicon mono-crystalline wafers and lithography machine components constitute as one of those sources and the wear characteristics are investigated using in-situ scratch experiments.

To resemble the wafer-machine contact situation at the asperity level, an extensive surface topography study has been performed using Atomic Force Microscopy (AFM) on the equipment that mechanically interacts with the silicon wafer. It was found that a sphero-conical indenter with a tip radius of 1 to 3 micrometer closely resembles the critical surface asperities. A range of scratch tests with these tips performed with Bruker's Hysitron PI 89 indeed replicate the wear particle behavior similar to that of chip formation on a lathe, demonstrating the influence of phase transformation through ductile behavior. Next, a wear volume study has been performed to calculate the effective loss in volume due to scratching and the relation to plasticity and wear particle generation. A numerical scratch model for mono-crystalline silicon has been developed that is currently being validated against the scratch test data.

11:45 "Micromechanical in-situ nanoXCT studies to examine fracture failure mechanisms of on-chip interconnect stacks"

Stefan Weitz[1,*], André Clausner[1], Ehrenfried Zschech[2]

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Semiconductor industry is continuing the downsizing of device structures and also of on-chip interconnect dimensions, both from performance reasons but also from economic reasons. This development has implications for the design of guard ring structures, i.e. metallic non-functional structures in the back end of line (BEoL) stack at the periphery of these microchips. On the one hand, these protective structures have to be designed to be efficient to stop microcracks and, on the other hand, the footprint of these structures has to be as small as possible. To test the performance of these guarding structures, micromechanical experiments are required in conjunction with appropriate characterization techniques to initiate crack propagation and enable its visualization. Thus, a novel micromechanical in-situ experiment was integrated into an X-ray microscope (ZEISS Xradia 800 Ultra). This experimental setup enables high resolution imaging of the 3D-patterned sample structures and defects in 2D and 3D with a resolution of up to 50 nm during in-situ testing. Performing tomographies allows non-destructive 3D visualization of the interior throughout the entire experiment and thus seamless 3D tracking of the microcrack. A customized sample geometry was developed which allows the application of a tensile load to a BEoL specimen by a lever mechanism. The lever is actuated by a microindenter whose indentation force is quantified simultaneously. In this work, mechanical degradation and failure mechanisms in a BEoL stack are studied, to gain understanding of the mechanical robustness of the pure interconnect structure.

12:00 "Bruker Nanoindentation Instruments"

Sanjit Bhowmick[1,*], Eric D. Hintsala[1], Jasmine Johnson[1], Douglas D. Stauffer[1]

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In-situ nanomechanical testing at cryogenic to elevated temperatures provides an opportunity to researchers to understand mechanical performance of materials that are exposed to extreme environments. The materials that are used in such applications include but are not limited to thin films, composite materials, superalloys, steels, high entropy alloys, nuclear materials, and micromechanical devices. Cryogenic-temperature environments often cause a severe deterioration in ductility and toughness of these materials by reducing damage-tolerance capacities, whereas in general, elevated temperatures lower the strength and increase plasticity. This presentation will highlight recent development of the in-situ instruments for testing materials at elevated and cryogenic temperatures.

Talks — Session II

Chair: Holm Geisler, GlobalFoundries Dresden Module One LLC & Co. KG

13:30 "Surface integrity on ground WC-Co hard metal grades under service-like working conditions"

J.J.Roa[1,*], G. Riu[1], M.A. Monclús[2], J.M.Molina-Aldareguía[2]

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The unique combination of hardness, toughness and wear resistance exhibited by heterogeneous hard materials (e.g. PcBN systems, cemented carbides, among others) has made them preeminent material choices for extremely demanding applications, such as metal cutting/forming tools or mining bits among others, where improved performance together are required. This presentation is focused on WC-Co cemented carbide material surface integrity evolution at high/intermediate temperature. A systematic micro and nanomechanical study of several WC-Co (in terms of metallic binder content and WC particle size) ground materials is presented. In general, three different aspects are investigated to accomplish the main goal of this research: (1) assessment of the intrinsic hardness of the deformed layer from room temperature up to 600ºC, (2) correlation of the compressive residual stresses with the hardness and elastic modulus maps by high indentation speed tests at the different temperature tested and (3) evaluation of the damage and plastic deformation mechanisms induced in the deformed layer. It was found that for different WC-Co composites, the intrinsic hardness of the deformed layer starts to slightly decrease at temperatures ranging between 400 and 500ºC, depending on the amount of metallic binder. This effect is attributed to different effects which take place simultaneously when the testing temperature increases: dislocation motion, reduction of compressive residual stresses and layer oxide generation mainly constituted by $CoWO_4$, WO_3 and Co_3O_4.

13:50 "Fracture toughness of cementitious composites assessed via nano-scratch"

Jiří Němeček[1,a,*], Radim Čtvrtlík[2], Vít Šmilauer[1], Jiří Němeček[1,b]

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Cementitious composites are the most used materials in the construction industry. At the microscale, it is a highly heterogeneous material consisting of several hydration products with different mechanical properties. These products are responsible for the overall mechanical performance of the composite at the macroscale. Assessing the local fracture properties of the products by standard nanoindentation is a challenging task due to the inaccessibility of the fracture surface, which is crucial for evaluation. A direct approach consists of bending of a micro-cantilever prepared by focused ion beam. Despite many advantages, this approach is very time-consuming and costly. An alternative, nano-scratch, presents a convenient method to access the fracture toughness of individual hydration products in a short time and with sufficient statistics. For gaining reliable results, support from other techniques, such as scanning electron microscopy (SEM) or acoustic emission (AE), is required. AE detects elastic waves during phase cracking. In the case of cement paste, cracking of only one specific phase (Portlandite) is detected, which significantly helps with the phase separation process. Moreover, a 3-D finite element model of the scratch process utilizing a fracture damage model with tension/compression failure can be successfully used to replicate the experiment until the first crack appearance. The model enables prediction of the crack depth and apparent tensile strengths of the products.

14:10 "Examining local fracture toughness of grain size tailored WCu nano-composites"

Klemens Schmuck[1,*], Markus Alfreider[1], Michael Burtscher[1], Michael Wurmshuber[1], Daniel Kiener[1]

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To assess fracture mechanical characteristics such as fracture toughness of materials, detailed assessment of the crack growth is essential. In the current work, the grain size influence on the local fracture characteristic of nanostructured WCu was investigated. WCu samples, made of 80 wt.% W and 20 wt.% Cu, were subjected to grain refinement by high-pressure torsion. To tailor the grain size of the composite in the nano-crystalline regime, the deformation temperature was varied between RT and 400°C. Microstructural saturation was verified by Vickers hardness measurements and SEM investigations. The later revealed a bi-modal microstructure, containing statistically distributed W inhomogeneities embedded in a nano-crystalline matrix. Additionally, STEM investigations were performed to analyze the matrix grain size, showing a clear relation between deformation temperature and grain size. To characterize the fracture behavior, micro-cantilever bending beams were fabricated by FIB milling. The cross-section of the cantilevers was varied from (5x5) μm^2 to (35x35) μm^2 to study potential sample size effects on fracture toughness. The tests were performed in-situ inside an SEM by applying quasi-static loading with partial unloading and enhanced by digital image analysis. For the smallest cantilever size, the analyzes revealed a slight increase of the fracture toughness, confirming presence of a sample size effect on the deduced fracture toughness. For all larger specimen dimensions, fracture mechanical evaluation revealed an achievable fracture toughness of about 10 MPa√m. Inspection of the fracture surface showed that intercrystalline fracture dominates the fracture process, and the inhomogeneities present in the nano-crystalline matrix govern the achievable fracture toughness.

14:25 “Micro- and meso-mechanical fatigue testing – abnormal grain growth, crack initiation and growth”

Jutta Luksch[1,*], Aloshious Lambai[2], Gaurav Mohanty[2], Florian Schäfer[1], Christian Motz[1]

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Fatigue mechanics is an important area in the design of a component because even with thermal cycling, a component can fatigue due to thermal expansion. With the development of methods to test even small volumes in recent decades, fatigue has become more important due to advances in test equipment. The study outlines the challenges of specimen and microtool fabrication. It also presents the test and analysis procedure for meso-mechanical fatigue. In most cases, specimens are fabricated from bulk material using FIB technology. In the current study, cantilever specimens are fabricated from nanocrystalline nickel with cross sections ranging from 5 x 5 to 50 x 50 microns. The large specimens cannot be fabricated in a reasonable time with the widely used Ga FIB, a Xe PFIB and for the medium sized specimens a combination of Ga and Xe FIB was used. The notched cantilevers are then object to fatigue loading with a negative load ratio to induce a pre-crack. That procedure is followed by the fatigue loading with a positive load ratio to collect crack growth data. A study of the fatigue crack growth rate, crack initiation and a study of grain growth in the crack region are discussed. Emphasis is placed on microstructural investigations using FIB and EBSD in the region of the crack. Here curtaining due to crack flanks is a challenge and the different specimen sizes make comparable microstructure analysis difficult.

14:40 “Measuring interface adhesion through nanoindentation and nanoscratching”

Shatha Almarri[1,*], Ed Darnborough[1], Dave Armstrong[1]

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Solid state batteries are regarded as the next improvement in battery technology due to their superior safety, energy density and lifetime. However, the replacement of the liquid electrolyte with a solid one, introduces complexities at the electrolyte-electrode interface. As the battery cycles and undergoes large volume changes, insufficient adhesion in this interface can lead to delamination which can greatly hinder lithium-ion movement and battery lifetime.

In this study two methods, nanoindentation and nanoscratching, are used to characterize the electrode-electrolyte interface and extract the strength of adhesion. A Berkovich indenter has been used in conjunction with Bruker's TI Premier nanoindenter. Scratch testing has generally been limited due to its high parameter sensitivity which complicates data interpretation. A study of how these parameters impact the observed critical load has been carried out. From the obtained critical loads, values of adhesive strengths have been calculated. Furthermore, delamination energy has been extracted through quasi-static indents by observing the deviation in the load-displacement curve. Focused ion beam (FIB) has been used in order to verify that delamination has taken place. The forces involved in nanoindentation and nanoscratching differ, therefore it is expected that the two tests give different values of delamination energy, and these are compared in this study.

14:55 "Tracking the evolution of local elastic strain fields in tailored metallic glass composites during in-situ deformation in the TEM"

Simon Fellner[1,*], Jürgen Eckert[1,2], Christoph Gammer[1]

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Despite having unique attractive properties, non-crystalline/amorphous materials suffer from catastrophic failure after reaching their yield strength. The ability to improve the mechanical and functional properties of metallic glasses depends on understanding their inherent strain state. Recently, nanoscale strain mapping was used to unravel shear band formation in a monolithic metallic glass [1]. The effect of structural changes induced by the introduction of secondary crystalline phases, highly rejuvenated regions or other defects renders transformation mechanisms in metallic glasses even more complex. Due to the emergence of novel dedicated specimen holders, in-situ TEM measurements are a powerful tool allowing to directly measure the deformation behavior of these tailored nanocomposites [2]. In this work, we track the evolution of local elastic strain fields in metallic glass composites during in-situ deformation simultaneously in amorphous and crystalline domains. To quantitatively measure local elastic strains at the nanoscale, we use a 4D-STEM setup incorporating an in-column energy filter in combination with a fast pixelated direct electron detector. This allows us to follow the structural response during deformation. As a result, a direct link can be established between the structure and properties of these tailored metallic glass composites.

The authors gratefully acknowledge the financial support from the Austrian Science Fund (FWF): Y1236-N37.

[1] H. Sheng, D. Şopu, S. Fellner, J. Eckert, C. Gammer, *PHYSICAL REVIEW LETTERS* 128 (2022) 245501.

[2] H. Sheng, et al. *Materials Research Letters*, 9 (2021) 190-195.

Talks — Session III

Chair: Mathilde Laurent-Brocq, Institut de Chimie et des Matériaux Paris Est, UPEC-CNRS

15:40 "Mechanical property changes accross the spin transition in a molecular film probed by nanoDMA"

Maryam Nasimsobhan[1], Lijun Zhang[1], Yuteng Zhang[1], Baptiste Martin[1], Gabor Molnar[1,*], Azzedine Bousseksou[1,*]

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The possibility of harnessing useful work from bistable molecular has led to the development of hundreds of compounds with a remarkable degree of sophistication. Here, we have shown that spin-crossover molecules of [Fe(HB(1,2,4-tryazol-1-yl)3)2] (compound 1), processed as thin films can be used to fabricate MEMS devices; demonstrating good mechanical integration of the molecules to produce useful work under a controlled external stimulus. The device also exhibits spin-state dependent resonance frequency and enhanced mechanical damping around the spin-transition temperature. To enhance the scope of these results, a detailed quantitative analysis of the mechanical properties and actuating performance of films of 1 becomes necessary. Viscoelastic property mapping by AFM revealed significant softening at the spin transition. For a more quantitative assessment of this phenomenon, we conducted a variable temperature nanoindentation study on a ca. 1.3 µm thick film of 1 deposited on a silicon substrate. Continuous stiffness measurements (CSM) of the film give access to the mechanical properties through the depth of the sample, providing with a tool that allows us to correlate mechanical properties to indentation depth and eliminate substrate and roughness effects. we conducted a nanoDMA study of the film between 20°C and 95°C with steps of 3°C. The results reveal a pronounced and well-reproducible variation of the sample mechanical properties at the spin transition around 60°C. This is characterized by drop in the storage modulus from 8.18 GPa to 4.16 going from low temperature (low spin) to high temperature (high spin) state.

Invited Talk

15:55 "Novel approaches to avoid FIB-artifacts in microcantilever fracture tests"

Subin Lee[1,*], Eloho Okotete[1], Yinxia Zhang[1], Steffen Brinckmann[2], Christoph Kirchlechner[1]

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Focused ion beam (FIB) based small-scale fracture testing has been well established in recent years. Simple cantilever-based geometries are the most prominent geometries used for micro-fracture studies of not only structural materials but also functional materials, such as thin films or hard coatings. However, the applicability of small-scale fracture testing for extracting bulk-like fracture properties is controversially discussed. A major source of concern is the presence of FIB-induced artefacts such as residual stresses due to ion implantation, chemical interactions of gallium ions including segregation at the notch tip, or finite size of notch root radius. In this talk, we demonstrate two novel approaches which can minimize FIB-artefacts in micro-cantilever fracture tests using atomically sharp natural cracks. Firstly, a new geometry for testing interface toughness at the micron scale will be presented. The geometry is straightforward to fabricate using FIB and enables observation of stable crack growth along a film/substrate interface. The other approach is using a well-defined thin bridge notch for a single cantilever fracture test. During in situ SEM fracture test, we directly observed that extremely thin bridges fail first, generating sharp natural cracks ahead which are free from FIB-artefacts. As a result, the fracture toughness of materials can be measured more precisely with less scatters. Furthermore, from the load drops at bridge failures, additional data points of fracture toughness can be obtained which is a huge advantage for statistical analysis.

16:25 "Mapping strain fields in CuZr-based metallic glasses by 4D-STEM assisted in-situ mechanical testing"

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Metallic glasses (MGs) are a promising new category of materials as they have great corrosion and wear resistance and high strength and hardness. However, they face the problem of being inherently brittle at room temperature and fracture immediately after yielding, making them unsuitable as structural materials. The deformation of MGs is characterized by strong strain localizations in shear bands, and rapid propagation thereof. Introducing heterogeneities in the form of secondary phases into the glassy matrix has proven to be a promising route to overcome this limitation. Here, we aim to directly map the elastic strain distribution in metallic glasses during in-situ deformation in the TEM. Monolithic $Cu_{50}Zr_{50}$ and multilayered $Cu_{64}Zr_{36}$-$Cu_{50}Zr_{50}$ thin films were synthesized via magnetron sputtering. Multiple bending beams were prepared via focused ion beam milling and subsequently tested in-situ in a scanning transmission electron microscope. The mechanical testing was performed using a Hysitron PI 95 from Bruker in displacement-controlled mode. We used nanobeam electron diffraction (NBED) to map the strain states with nanometer spatial resolution. By pausing the deformation experiment at multiple points, the temporal evolution of the strain states could be captured. The results show a significant reduction in the size of the elastic strain field surrounding the notch in the composite material. In the case of the multilayered samples, the strain was mainly concentrated inside the first layer, while the extend of the highly strained region in the monolithic sample was far larger.

16:40 “Initial Analysis on the Statistics of Serrations and Strain-Rate Sensitivity of Vit105”

Valeria Lemkova[1,*], Tobias Thielen[1], Florian Schäfer[1], Christian Motz[1]

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If amorphous metals are plastically deformed, in comparison to crystalline materials, no dislocations can form which can counteract the local stress state. Instead, so-called shear bands develop. This is determined by the load level, deformation speed, stiffness, and the available material volume but also, by the free volume. These volumes describe the necessary free area in a rearrangement process of atoms or clusters. It can be adjusted via a specific temperature control. If a POP-in event occurs during nanoindentation of amorphous metals, such a shear bands are now introduced into the material. With CSR (constant strain rate) tests, these POP-ins were statistically evaluated, and serration lengths were determined. different strain rates and set free volumes were compared for Vit105 ($Zr_{52.5}Cu_{17.9}Ni_{14.6}Al_{10}Ti_{5}$) [1-3].

[1] Yang, B., et al., *Acta Materialia* 55.1 (2007): 295-300. https://doi.org/10.1016/j.actamat.2006.08.028

[2] Cheng, L., et al., *Journal of Applied Physics* 115.8 (2014): 084907. https://doi.org/10.1063/1.4866874

[3] Neuber, N., et al., *Scientific Reports* 12.1 (2022): 17133. https://doi.org/10.1038/s41598-022-20938-6

Keynote Lecture

Chair: Christian Motz, Saarland University

19:00 "Probing defect - defect interactions in metals and (complex) alloys by in-situ nanomechanics"

Prof. Dr. Gerhard Dehm[1,*]

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Defects are omnipresent in most materials and decisive for their mechanical properties. The extension of nanoindentation based methods in the last decades for in-situ studies stimulated new insights on the kinetics and evolution of defects like dislocations and their interactions with point defects, dislocations and grain boundaries. In this overview presentation, three examples will be provided on studying dislocation interactions with near atomic pinning sites. The first part of the talk will focus on "high entropy alloys", where for the FeCoCrMnNi alloy ("Cantor alloy") conflicting results concerning the presence of short range ordering and its impact on plasticity are reported in literature. The results of micro-pillar compression testing indicate differences in the size scaling exponent compared to pure fcc metals. However, only the combination of small scale mechanical testing, in-situ and post-mortem transmission electron microscopy together with atomistic simulations provide evidence that even in a Cantor alloy without short range ordering, the random atomic neighborhood can create strong fluctuations in the local Peierls stress, which explain the dislocation pinning events [1].

In the second case study the interaction of dislocations with hydrogen is investigated for FeCr alloys using a new electrochemical cell design integrated into a nanoindenter. The set-up allows to charge the specimen from its backside [2] leaving the front side unaffected from unwanted corrosion effects. Our recent results show reproducible effects on the hardness and strength evolution during in-situ nanoindentation and micro-pillar compression. Furthermore, we could also reveal that in-situ nanoindentation is sensitive enough to measure the diffusion coefficient of hydrogen in the FeCr alloys. The diffusion results were verified by Kelvin probe measurements.

Finally, we studied dislocation transmission through twin boundaries in Cu. While it is known that screw dislocations can cross a twin boundary, there are controversial data available on the required penalty stress for transmission. Molecular dynamics simulations suggest relatively high stress values, but are of course limited by high strain rates. With micropillar compression testing using different pillar sizes, we can probe dislocation-based models for transmission. Indeed, the penalty stress required for easy dislocation transmission of a twin boundary is not a constant transmission stress, but dominated by dislocation curvature of non-screw segments traversing the twin boundary [3]. All three examples show that in-situ small scale mechanical testing has advanced into a quantitative tool for probing defect-defect interactions especially in combination with microscopy techniques and atomistic simulations.

[1] Utt, Daniel, et al. "The origin of jerky dislocation motion in high-entropy alloys." *Nature Communications* 13.1 (2022): 4777.

[2] Duarte, Maria Jazmin, et al. "In situ nanoindentation during electrochemical hydrogen charging: a comparison between front-side and a novel back-side charging approach." *Journal of Materials Science* 56 (2021): 8732-8744.

[3] Hosseinabadi, Reza, et al. "Size scaling in bi-crystalline Cu micropillars containing a coherent twin boundary." *Acta Materialia* 230 (2022): 117841.

Abstracts - Oral Presentations

Day 2: Wednesday, May 24th

Talks — Session IV

Chair: Claudia Fleck, Technische Universität Berlin

Invited Talk

09:30 "Nanoprobing water inclusive materials"

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The effect of water during nanoindentation is usually not considered, but it plays an important role both at probe contact and also during deformation of water-laden materials. We have undertaken initial studies on the measurement of water layer thickness at subzero temperatues and will show the interaction of the probe with water during this test. Continuing studies will address the measurement of friction on ice at subzero temperatures. A new test is being developed for looking at ice friction that requires alternative probe design. Probe design will be explained to show the extended scope of application for Bruker indentation tools. The needs of mechanical testing in water-laden foods will be explained to further improve the eating experience.

10:00 "Humidity-Dependent Mechanical Properties of Halide Perovskites"

Sidney R. Cohen[1,*], Irit Goldian[1], Isaac Buchine[2], Davide R. Ceratti[3,4], Naga Prathibha Jasti[2], Sujit Kumar[2,3], David Cahen[2,3]

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Halide Perovskites (HaP) demonstrate exceptional promise for photovoltaic applications, but are known to undergo degradation upon exposure to ambient humidity. Mechanical properties of these materials can influence the dynamic process of ion migration, as well as non-radiative recombination at strain-induced defects. These facts underline the need to understand the influence of humidity on mechanical and structural properties of these materials. Here, atomic force microscope (AFM) and instrumented nanoindentation (INI) mechanical studies were performed on five different HaP single crystals of formula ABX_3:
A= Methylammonium (MA), B= Pb, X=Chloride (Cl), Bromide (Br), or Iodide (I), and B=Pb, C= Br, and A= Formamidinium (FA), or Cesium (Cs). Modulus and hardness of the materials were measured at low (5-10%) and ambient (55-60%) relative humidity. Striking differences in the humidity-dependent mechanical properties of the different crystals could be rationalized through structural and electrostatic considerations. These differences were also reflected in the difference between bulk (INI) and surface (AFM, measured by contact resonance mode) values of the modulus. We found that exploiting the strengths of each technique gave interesting insights on the mechanical response of these crystals. In particular, comparison of hardness computed through Oliver and Pharr analysis to "traditional hardness" using the directly measured final imprint area was critical to interpreting our observations.

10:20 "Evolution of Mechanical Strains in Biological Ceramics"

Shahrouz Amini[1,*], Tingting Zhu[1], Erika Griesshaber[2], Peter Werner[1], Peter Fratzl[1]

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Quantitative understanding of the micromechanical behavior of biological ceramics, in which anisotropies, heterogeneities, and interfaces are omnipresent, requires methodological approaches allowing measurement of the mechanical strains during their development. Here, we introduce a devised method by which elastic strain fields (ESFs) can be mapped in operando, under contact loads, in 3D, and with sub-micron spatial resolution. We deploy our method to showcase the development of ESFs in biological ceramics and to compare them with their geological counterparts. We show how the presence of the crystallographic organization and organic inclusions influence the formation and development of ESFs, and we unravel how ESFs can regulate the formation of inelastic deformations.

10:40 "Exploring the micromechanics of the strongest known biomaterial: How the limpet tooth realizes a higher strength than hardness"

M. Wurmshuber[1,*], S.H. Oh[2,3], J.-K. Kim[2,4], Y. Liu[5,6], X.-L. Peng[7,8], J. Seo[2,3], J. Jeong[2], Z. Wang[2], J. Wilmers[7], C. Soyarslan[7,9,10], J. Kim[2,3], B. Kittiwirayanon[2], J. Jeong[2,3], H.-J. Kim[11], Y.H. Huh[11], S. Bargmann[7,12], H. Gao[5,13,14], D. Kiener[1]

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In order to feed themselves, limpets – a group of marine snails – use their radula full of microscopic teeth to scrape algae from rocks. Therefore, it is to no surprise that these teeth have recently been found to be one – if not the – strongest materials in nature. Extensive microstructural and micromechanical characterization of limpet teeth has been performed and is presented herein. It was found that the more heavily stressed leading part of the tooth consists of iron-oxide nanorods embedded in a silica matrix. Some of these rods are assembled into bundles and orientated perpendicular to the rest of the rods. These nanorod bundles exhibit multiple rotational movements, which ultimately lead to a local auxetic behavior of the composite microstructure, likely a major reason for their extraordinary strength. Using micropillar compression testing, we could confirm the high strength of over 3 GPa reported in earlier work and also demonstrate an overall Poisson's ratio of close to zero for the limpet tooth composite. Moreover, the measured strength was found to be higher even than the hardness gained from nanoindentation. Using micromechanical FEM modeling, this peculiar phenomenon is explained by microdamage formation in-between the auxetic nanorod bundles as a consequence of the sharp indenter tip, high indentation strain and local tensile strains from auxetic rotation.

11:00 "Compression of pellets from filamentous microorganisms"

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Filamentous microorganisms are widely used in industrial biotechnology as production systems for e.g. active pharmaceutical ingredients or organic acids. Depending on the cultivation conditions, biomass growth occurs as dispersed mycelium, clumps or dense bioagglomerates - also called pellets. In stirred bioreactors, the morphology is mainly determined by the shear stresses induced by the fluid.

To characterise the mechanical properties of whole pellets, a micro-compression test has been established using the Bruker TriboIndenter. The compaction test can be visually monitored and the shape of the pellet assessed using an additional camera. With this test set-up, multiple loading, creep and relaxation tests were carried out in addition to single compression tests.

The results show visco-plastic properties that could be modelled using Maxwell elements.

In addition, the pellet structures were simulated by a growth model as a digital twin and using the discrete elements method (DEM).

Talks — Session V

Chair: Subin Lee, Karlsruhe Institute of Technology

Invited Talk

11:50 "Nanoindentation on Microchips: Thermomechanical Characterization beyond Hardness and Reduced Modulus"

Holm Geisler[1,*], Dirk Breuer[1], Petra Hofmann[1], Frank Küchenmeister[1]

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Nanoindentation (NI) with all its additional advanced features, like lateral load detection and sensitive dynamic-mechanical analysis, is a versatile tool to investigate local mechanical properties on microelectronic chips on wafer level with high spatial resolution, and in the meantime also over a wide temperature range. The portfolio of NI based techniques at GlobalFoundries includes methods like pillar shear testing to mimic CPI loading conditions on copper pillar bumps, temperature dependent NI (-100°C to 400°C), nanoDMA, and nano-scratch testing for thin film adhesion assessment. In the semiconductor industry, the materials analyst is facing a wide variety of materials, like hard and brittle thin films (e.g., SiN), metals (e.g., Cu, Al, solder) with sometimes considerable creep behavior and various microstructures, polymers (like polyimide), and micro-porous ultra-low k dielectric films. In order to assess the overall mechanical reliability, e.g., with respect to chip-package interaction, quantities beyond reduced elastic modulus and hardness must be analyzed. These are (1) local fracture toughness of (patterned) thin films or film stacks, (2) yield strength, (3) strain rate sensitivity, and (4) creep characteristics, viscoelasticity or viscoplasticity. The qualitative comparison of the fracture behavior of different passivation film stacks by means of an empirical NI approach will be presented. Beyond that, an attempt is made to quantify the fracture toughness. It will be shown that differences in the crack behavior of various film versions are detected in this way. Secondly, the temperature dependent creep behavior of polyimide films and solder materials are investigated and analyzed using the effective indenter concept and reference creep testing. The purpose of these measurements is to find suitable parameters for the definition of the constitutive equations of a small volume of such materials.

12:20 "Characterization of nanomaterials using On-Axis TKD in SEM: challenges and benefits"

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Transmission Kikuchi Diffraction (TKD) in a Scanning Electron Microscope (SEM), a.k.a. transmission EBSD (t-EBSD) has been first proposed in 2012 and has quickly become an established technique due to its much better spatial resolution compared to Electron BackScatter Diffraction (EBSD). The initial TKD sample-detector geometry was optimized a few years later by the introduction of "On-Axis TKD" leading to major gains in spatial resolution, data acquisition speed, and data integrity. These improvements have helped expand the applications range of TKD in SEM by enabling quantitative and qualitative characterization of nanomaterials only possible before in a Transmission Electron Microscope (TEM).

The talk will present recent further advancements in spatial resolution limit and data quality, enabled by hardware and software developments. Efforts to automate, time resolved mapping and imaging of nanomaterials and thin films during dynamic / in-situ experiments, will also be explored. Challenges ahead in the process of making On-Axis TKD technique a robust, state-of-the-art tool for nanomaterials and beam sensitive materials research will be reviewed. The discussion will be supported my multiple experimental results acquired from various materials and experimental conditions.

Invited Talk

12:40 "Mechanical Properties of 2D-like Polydopamine Films"

Emerson Coy[1,*]

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Polydopamine (PDA) is a bioinspired and versatile polymer with numerous applications in various fields, including electronics and biomedicine. Its unique properties, such as adhesive, catalytic, and biocompatibility, make it a popular material for many researchers looking for a relatively straightforward and inexpensive polymer. In recent years, the development of large-scale, free-standing thin films has attracted significant attention in the scientific community, which has quickly expanded this interest to polymeric materials. Generally, PDA thin films can be prepared using methods such as solution-casting, Langmuir-Blodgett, or spray-coating techniques. However, producing large-scale free-standing films with consistent quality and mechanical properties is challenging. Moreover, the mechanical properties of such films have been explored in several studies, with scattered and discouraging results. Despite this, quite recently, we have studied the physicochemical properties of thin PDA films, from the air/water interface, with an exceptional mechanical response [1]. We have also shown outstanding control over their thickness and morphology [2]. Therefore, in this presentation, I will present the general status of the preparation of large-scale free-standing PDA thin films, their structure and, more importantly, their mechanical properties as a general indicator of their internal structure.

[1] E. Coy, I. Iatsunskyi, J. C. Colmenares, Y. Kim, R. Mrówczyński, *ACS Appl. Mater. Interfaces* **2021**, 13, 23113.

[2] J. Szewczyk, M. Pochylski, K. Szutkowski, M. Kempiński, R. Mrówczyński, I. Iatsunskyi, J. Gapiński, E. Coy, *Mater. Today Chem.* **2022**, 24, 100935.

Talks — Session VI

Chair: Gerhard Dehm, MPI für Eisenforschung and Ruhr University Bochum

Invited Talk

14:30 “Nanoindentation, a powerful tool to study strengthening in high entropy alloys”

Mathilde Laurent-Brocq[1,*], Guillaume Bracq[1], Diaa Mereib[1], Ivan Guillot[1], Jean-Marc Joubert[1], Céline Varvenne[2], Judith Monnier[1], Loïc Perrière[1], Benjamin Villeroy[1]

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Since the discovery of the single-phased CoCrFeMnNi alloy and the proposition of the concept of high entropy alloys (HEA) in 2004, it appears that the interest of HEA does not lie in their specificity but rather in the opportunity that they raise to explore wide domains of composition and, hopefully, to find unprecedented mechanical properties. To do so, nanoindentation is a powerful tool.

First, the concept of high entropy alloys will be presented. Then the thermodynamic stability and the solid solution strengthening will be explored in the entire quinary Co-Cr-Fe-Mn-Ni system [1, 2]. We will show how nanoindentation was used to measure hardness on a wide range of chemical composition and then to validate a solid solution strengthening model. Here the useful advantage of nanoindentation was its ability to perform measurements on small samples. Finally, high entropy alloys have inspired a new strengthening mechanism, by chemical architecturation. After briefly exposing the processing and the microstructural characterization, we will show how nanoindentation was determinant to prove the novelty of this strengthening effect [3, 4]. Here the useful advantage of nanoindentation was the submicronic scale and the quantitative measurements.

[1] Bracq, G., et al., The fcc solid solution stability in the Co-Cr-Fe-Mn-Ni multi-component system. *Acta Materialia*, 2017. 128: p. 327-336.

[2] Bracq, G., et al., Combining experiments and modeling to explore the solid solution strengthening of high and medium entropy alloys. *Acta Materialia*, 2019. 177: p. 266-279.

[3] Laurent-Brocq, M., et al., Chemical architecturation of high entropy alloys through powder metallurgy. *Journal of Alloys and Compounds*, 2020. 835: p. 155279.

[4] Mereib, D., et al., Chemically architectured alloys: How interphase width influences the strengthening. *Journal of Alloys and Compounds*, 2022. 904: p. 163997.

15:00 “Development of novel nanoindentation-based stress relaxation tests to study transient plasticity in metals”

Gaurav Mohanty[1,*], Suprit Bhusare[1], Aloshious Lambai[1], Jakob Schwiedrzik[2], Johann Michler[2]

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Nanomechanical tests are moving beyond regular hardness and modulus measurements to encompass host of different mechanical properties like strain rate sensitivity, creep, and fracture toughness. Developing the capability to measure stress relaxation behavior of materials will be useful to not only extend the gamut of properties studied at the micro/nanoscale, but also to study fundamental time-dependent deformation behavior of materials. This poster will present, for the first time, the development of indentation-based stress relaxation tests that can reliably extract the activation parameters for deformation at micrometer length scales. The developed testing methodology, data analysis framework and the validation results on nanocrystalline metals will be presented. Nanocrystalline metals were selected for validation tests because they do not show size effects in indentation since hundreds of nanometer sized grains are interrogated in the indentation zone. Therefore, the micromechanical response can be considered to be “bulk” even in case of these small-scale tests. The indentation results were systematically compared with compression tests on micrometer sized pillars which have previously been shown to exhibit “bulk” scale behavior. A good match was found between both indentation and micropillar compression tests validating the developed testing methodology. This opens the possibility to perform transient stress-relaxation tests on thin films and individual grains in future to systematically study the operative deformation mechanism(s) in a wide range of materials.

15:20 “Revealing deformation mechanisms by scale-bridging mechanical testing from in situ electrochemical nanoindentation to strain rate jump tensile tests”

Florian Schaefer[1,*], Alexander Erke[1], Rouven Schneider[1], Christian Motz[1]

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The strain rate sensitivity of a material arises from a thermally-activated contribution to the deformation processes, especially to dislocation slip. For instance, in nanocrystalline f.c.c. metals, a multitude of grain boundaries forcing the dislocations to interact with them during plastic deformation causes the strain rate sensitivity to increase. In the following study, the extent to which hydrogen affects thermal-activated dislocation mobility and thus strain rate sensitivity was investigated in nickel and ferritic perlitic steel. For this purpose, specimens were charged in situ, both cathodically and by low-pressure hydrogen plasma, and subjected to nanoindentation, micro-pillar compression, and strain-rate jump macro-tensile tests, and the results were contrasted. Macro tensile tests were performed down to 195 K. Hydrogen is shown to increase the strain rate sensitivity of f.c.c. not of b.c.c. metals and affecting the deformation mechanisms itself. The rate-limiting deformation process is identified via the activation volume derived from the strain rate sensitivity.

15:40 “Detecting local hydrogen content in materials by scanning Kelvin probe force microscopy”

Christian Motz[1,*], Patrick Grünewald[1]

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In this talk methods for determining the local distribution of hydrogen in metallic materials are investigated and supplemented by micromechanical measurement methods. The focus is on the further development of the hydrogen detection method based on Scanning Kelvin Probe Force Microscopy. Its measurement signal correlates with the electrode potential of a metal-hydrogen electrode (and thus the concentration of hydrogen) which is formed in the system of sample, dissolved hydrogen and ambient humidity. The method is used and further developed in a separate in situ experimental setup to determine local hydrogen distributions at grain boundaries. In particular, the influence of the atmosphere (oxygen content and humidity) on the detected hydrogen is systematically investigated. Nanoindentation is additionally used to provide information on the local distribution of hydrogen by measuring mechanical properties on a small scale. Both measurement methods are performed successively on the same sample locations and the results about the local hydrogen distribution are compared with each other. The two measurement methods are supplemented by permeation tests and macroscopic and microscopic mechanical test series.

Talks — Session VII

Chair: Oden Warren, Bruker

16:30 “Multi-scale mechanical testing and high-resolution microstructural characterization: Investigation of deformation mechanics of thin hard coating”

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Comprehensive investigations of industrial wear-resistant thin hard coatings deposited on a hard metal through chemical vapor deposition (CVD) is presented. Mechanical testing (Pillar micro-compression, spherical contact damage) and microstructural characterization at different length scales (bulk, microstructural and atomic) were deployed to study deformation mechanisms of two cutting inserts systems consisting of a defined WC-Co cemented carbide substrate coated with two different CVD thin films: Ti(C,N) and Zr(C,N). The latter system exhibited a superior tool life in comparison to the conventional Ti(C,N). Several characterization techniques were used: confocal microscopy, scanning electron microscopy, focused ion beam, electron back scattered diffraction, X-ray synchrotron, atom probe tomography (APT) and high-resolution Secondary Ion Mass Spectrometry imaging (nano-SIMS). Unlike Zr(C,N) micro-compression of Ti(C,N) pillars revealed that the failure mechanism and crack initiation and propagation is exclusively through the grain boundaries [1]. The same result occurs at a larger scale with spherical contact damage tests which denote the intrinsic low cohesive strength at the Ti(C,N) interfaces [2]. High resolution characterization with APT and nano-SIMS revealed segregation of chlorine at the Ti(C,N) grain boundaries [3,4]. This finding is a relevant factor explaining the low cohesive strength at the grain boundaries of Ti(C,N) leading to exclusive intergranular failure.

[1] I. El Azhari, J. Garcia, M. Zamanzade, F. Soldera, C. Pauly, L. Llanes, F. Mücklich, Investigations on micro-mechanical properties of polycrystalline Ti(C,N) and Zr(C,N) coatings, *Acta Materialia*. 149 (2018) 364–376.

[2] I. El Azhari, J. García, F. Soldera, S. Suarez, E. Jiménez-Piqué, F. Mücklich, L. Llanes, Contact damage investigation of CVD carbonitride hard coatings deposited on cemented carbides, International Journal of Refractory Metals and Hard Materials. 86 (2020) 105050.

[3] I. El Azhari, J. Barrirero, J. García, F. Soldera, L. Llanes, F. Mücklich, Atom Probe Tomography investigations on grain boundary segregation in polycrystalline Ti(C,N) and Zr(C,N) CVD coatings, *Scripta Materialia*. 162 (2019) 335–340.

[4] I. El Azhari, J. Barrirero, N. Valle, J. García, L. von Fieandt, M. Engstler, F. Soldera, L. Llanes, F. Mücklich, Impact of temperature on chlorine contamination and segregation for Ti(C,N) CVD thin hard coating studied by nano-SIMS and atom probe tomography, *Scripta Materialia*. 208 (2022) 114321.

16:50 "Nanomechanical Investigations of Tribofilms Formed by ZDDP and Phosphonium-based Ionic Liquids on Bearing Surfaces"

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In terms of sustainability, the long and wear-free operation of rolling bearings is of high importance. To achieve a stable tribological system for lubricated contacts under mild wear conditions, the formation of a tribofilm is desirable. The additives in the lubricants have a great influence on the formation of a boundary layer. A typical antiwear additive is zinc dialkyldithiophosphate (ZDDP), which forms a smooth film with low roughness on the surface. Such a film consists of glassy Fe/Zn polyphosphates with a height of up to 150 nm. The film formation is favored by high shear stresses under high contact pressures and is additionally influenced by temperature. A relatively new variant for additives are ionic liquids (ILs). The tribofilm formation of two phosphonium-based ILs (trihexyltetradecylphosphonium bis(2,4,4-trimethylpentyl)phosphinate and trihexyltetradecylphosphonium bis(2-ethylhexyl)phosphate) is compared with a ZDDP-based tribofilm. The formation of phosphate-containing films under tribological contact was investigated on samples of 100Cr6 rolling bearing steel. The tribofilms formed were examined microscopically, by nanoindentation, and ToF-SIMS (Time of Flight - Secondary Mass Spectrometry). The investigations allow conclusions to be drawn about tribofilm formation in relation to phosphate and other element-containing layers. The model tests were compared with real bearing tests on an FE8 test sand. The results show that comparable tribofilms can be detected.

17:10 "Adaption of TI 950 for microfretting experiments"

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A trend in electrical contacts is miniaturization. Still, systems will always be exposed to vibrations, so that fretting contact conditions remain an issue. Here, we show the adaption of a Hysitron TI 950 Nanoindenter with an external piezo stage to measure the reaction of electrical contacts under vibrations in a load range of less than 100 mN to several Newton.

The evaluation of fretting behavior requires the analysis of friction loops, i.e. the friction force over displacement. The data synchronization of the external stage and the force measurements of the 3D OmniProbe was achieved by the integration of an additional DAQ board into the Performech-Controller. A second camera allows for the precise positioning of two contacts, when the classical tip-to-optics approach isn't possible due to sample geometries. Contact resistance measurements yield the crucial value for the evaluation of the lifetime of the electrical contact.

With the described adaptions, microfretting experiments of electrical contacts are possible. An exemplary result is shown.

Abstracts - Poster Presentations

1. "Calo Indent and Droplet Erosion Module – New methods to evaluate stress distributions and profiles"

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Proper characterization and optimization of complex structures requires invertible mathematical tools of sufficient holistic character. SIO developed analytical models which dramatically speed up the simulation of complex contact situations compared to FEM systems.

Together with sophisticated measurement devices it's now possible to characterize such complex material stacks. Knowing the material parameters of all parts allows to perform and analyse more complex experiments like scratch tests. This will give a view inside the material to really see what happens during a contact experiment and it will make it easier to find the initial failure.

In many deposition processes intrinsic (or residual) stresses can't be avoided during the coating creation. Mostly because of bias or high deposition temperatures and the mismatch in the coefficients of thermal expansion for the various materials. The intrinsic stresses can have a big influence on the material behaviour in contact situations.

In one way they can help fighting against your critical external loads and reducing the created stresses. On the other way they can also weaken the material compound when producing too much stresses in a weaker part of the system. Both sides can also have an effect on the adhesion strength between the different coatings. So gaining knowledge about these intrinsic stresses could help a lot in the field of modelling or simulating your worst case application scenarios.

This talk will show a new method to evaluate the stress profiles (e.g. yield strength and residual stresses) using extended indentation measurements and new mathematical models. The new method uses a reference probe with known intrinsic stresses and with new mathematical models the intrinsic stresses and more within your material can be evaluated.

In the second part this talk will present a new module which is currently under development. This module will be able to calculate the stress distribution created by particles and droplets during the contact. The droplet erosion module shall be able to evaluate the erosion caused by such impacts in a later development step. The module will take impact loads and the lateral movement of the droplets after the impact into account to predict the surface changes. Animations will show the actual state of development of the new module.

2. “PeakForce QNM, the pathway to interrogate nanomaterials”

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In the last years, much effort has been put into developing exfoliation methods for obtaining new two-dimensional (2D) materials. As a result, nowadays a huge variety of exfoliated 2D materials with different properties and applications are available. Further work in this area is the fine tuning of their properties through post synthetic routes. These procedures often involve the chemical or physical molecular functionalization of the 2D. This way it is possible to obtain some 2D flakes covered with molecules or even other 2D materials.[1]

Although this approach has enormous possibilities, frequently it is quite difficult to determine the success of the functionalization and its homogeneity.[2]

In this work, we take advantage of the spatial resolution of an atomic force microscope and the possibility to perform quantitative nanomechanics (QNM) with a Bruker-Icon AFM to obtain information related to the adhesion of functionalized and non-functionalized 2D layers of $MnPS_3$. As this method is based on the chemical nature of the sample it is possible to distinguish between two different 2D materials that have similar topography (size, shape, and thickness).

[1] Chen, Xin, and Aidan R. McDonald. “Functionalization of two-dimensional transition-metal dichalcogenides.” *Advanced Materials* 28.27 (2016): 5738-5746.

[2] Brotons-Alcázar, I., Torres-Cavanillas, R., Morant-Giner, M., Cvikl, M., Mañas-Valero, S., Forment-Aliaga, A., and Coronado, E. (2021). Molecular stabilization of chemically exfoliated bare MnPS3 layers. *Dalton Transactions*, 50(44), 16281-16289.

[3] Ito, T., Ibrahim, S., & Grabowska, I. (2010). Chemical-force microscopy for materials characterization. *TrAC Trends in Analytical Chemistry*, 29(3), 225-233.

3. “Investigations of the joining zone on hybrid profiles”

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Hybrid components made of different materials offer many advantages because, unlike conventional monolithic design, they allow for an adjustment of locally adapted properties. One manufacturing approach for hybrid components is investigated in the Collaborative Research Centre (CRC-1153) “Tailored Forming” by using pre-joined hybrid semi-finished products. In this context Co-extrusion is a possible process for the production of hybrid semi-finished products. However, in hybrid components as well as hybrid semi-finished products, joining zones necessarily occur in which the mechanical properties change abruptly. In the case of dissimilar materials such as aluminium and titanium or aluminium and steel, intermetallic phases are formed that are very hard and brittle compared to the base material. For this study, hybrid hollow profiles were produced using the co-extrusion process, in which different materials were surrounded by EN AW-6082 as a reinforcing element in the form of a tube. The reinforcement material used was titanium grade 2, titanium grade 5 and the bearing steel AISI 51200. The profiles are subsequently subjected to a heat treatment to increase the bond quality for the subsequent closed-die forging process. The influence of the heat treatment on the joining zone resulting from the co-extrusion process was investigated by means of nanoindentation. In order to evaluate the property changes in the joining zone due to heat treatment, large-area XPM measurements were performed. Performed measurements showed a hardness increase. The intermetallic phase grew with the heat treatment, increasing the quality of the compound.

4. "Mechanical properties of oxide scale under hot forming conditions on a nano level"

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Demands on the predictive accuracy of numerical analyses are increasing due to global factors such as resource conservation and advances in computational capacities. Consequently, the simulation of hot forming processes faces new challenges. One of which involves the consideration of oxide scale layers, as they strongly influence boundary conditions such as tribological properties and wear. On iron-based substrates, these layers form at temperatures above 570°C. In this context, scale growth is mainly influenced by temperature, process duration, and alloying elements. Three iron oxides are characteristic for steel: wustite (FeO) is the innermost oxide layer and forms the interface between scale and substrate, magnetite (Fe_3O_4) forms the intermediate layer and hematite (Fe_2O_3) is the outermost layer and in direct contact to the atmosphere. Within the scope of this work, nanomechanical characterisation (Hysitron TI 950 TriboIndenter) has been performed to identify mechanical properties of wustite, magnetite and hematite. In detail, hardness mappings and scratch analysis were performed to characterise the properties in cross-section of the oxide scale layers. For this purpose, specimens (45Cr4) were reproducibly oxidised using a dilatometer (Waters DIL 805A/D+T). Using XPM hardness mapping, it was possible to visualise phase transitions, interface damage and porosity of iron oxides in accordance to microscopy images. Based on these insights, future numerical analyses of oxide scale components can be modelled more accurately considering interface damage and depth dependent frictional properties.

5. "Experimental tips for reliable measurement of mechanical properties on hard coatings by nanoindentation"

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In this work, we discuss some experimental aspects of the Oliver and Pharr method that are often ignored and need to be considered to obtain reliable results. We evaluated the mechanical properties of Ti-Al-Si-N coatings grown on Si (100) and WC-Co. The indentation measurement on each substrate reveals specific challenges; on the one hand, the reduction of data dispersion by properly polishing rough samples will be demonstrated, and, on the other hand, the influence of size and gluing on Si substrates will be discussed. In addition, the fitting range of the unloading curve, and the depth range and age of the tip calibration area function will be evaluated. Finally, it is verified that each substrate shows a different influence on each property evaluated. Thus, the variation of elastic modulus with depth is different if the substrate is stiffer than the coating or vice-versa. In addition, it is shown that the optimal penetration depths to evaluate the elastic modulus and hardness are different. This indicates that the typical rule of '10% coating thickness' is only valid for 'local' phenomena, such as those involving hardness measurement. However, it is incorrect to properly evaluate elastic modulus, which deals with contributions at longer ranges. Therefore, hardness is better measured at higher depths, while elastic modulus should be evaluated near the substrate. In fact, more reliable values can be obtained by fitting the elastic modulus data at different indentation depths. A simple fitting function representing the stiffness transition from the coating to the substrate will be presented.

6. “Rose Prickles: Multiscale Structure-Mechanics-Function Relationships”

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Plant biomechanics is a major pathway in the field of biophysics that links the structures, materials, and the functions of plant organs to their mechanical loadings. Hook-like plant elements (spines, thorns and prickles) provide plants with various mechanical functions, such as defense, anchoring and frictional support. Identifying the multiscale mechanical and structural characteristics of these hook-like elements is essential to understanding their biomechanical adaptations to the function they serve. In this study, we analyzed the mechanical properties and the structural geometrics of the prickles of the dog rose by using nanoindentation techniques, micro-CT scans, and finite element simulations (FEA) across their nano-micro-macro structural levels. The results present in a new light the basic biophysical aspects of hook-like plant elements and introduce novel scientific-engineering outlines for the development of advanced functional materials for various biomedical and microengineering applications.

7. “Nanomechanical measurement of near-surface and interfacial properties for polymeric additive manufacturing materials”

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In past decades additive manufacturing (AM) technologies including 3D printing have shown promising potential such as tool-less manufacturing, rapid prototyping and mass customerization, and therefore gained more and more interest in various scientific and industrial fields. To date, AM technologies can well prototype not only mechanical components and devices, but also functional electrical components and systems, including passive resistors, capacitors, inductors, and even functional circuits [1]. In the meantime, reliable applications of AM-fabricated prototypes and products demand reliable quality control of these components made of polymer and metal powders, including quantitative determination of their mechanical properties.

In this contribution, quantitative nanomechanical characterization of the surface and interfacial properties of reference and AM materials are reported. Samples involved in the experimental investigation are a few typical bulk reference polymers such as PC, PA, Teflon and Peek coated with ~ 1 μm thick Au film, and one AM substrate made of typical PA powders with similar Au-coating. Nanoindentation technique is employed to investigate the depth-dependent mechanical properties of these materials. Finally, the near-surface mechanical properties of bulk materials and AM substrate with metallic coating have been analytically modelled and compared.

This research project is supported by the European Union and is funded within the scope of the European Metrology Programme for Innovation and Research (EMPIR) project 19ENG05 NanoWires entitled ‘High throughput metrology for nanowire energy harvesting devices’ (https://www.ptb.de/empir2020/%20nanowires/home/).

[1] A. H. Espera, J. R. C. Dizon, Q. Chen, and R. C. Advincula, “3D-printing and advanced manufacturing for electronics”, *Prog. Addit. Manuf.* 4, 245 (2019).

8. “The nanoindentation measurements influenced by surface topography”

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Nanoindentation is a method to assess the mechanical properties of thin films and bulk materials, which are among the crucial quantity required to their practical application. However, it is well known that when a sharp indenter for small penetration depths is employed, the surface topography of the investigated materials may influence the nanoindentation data. PECVD was used to deposit a thin a-CSi:H film with a thickness of 1.0 µm on a silicon wafer. The cyclic nanoindentation was chosen to obtain a depth profile of mechanical characteristics for the film’s flat surface (0.5 nm roughness), therefore, elastic modulus and hardness unaffected by the substrate was observed, namely 83 GPa and 8.6 GPa, respectively. The grains (spherical caps) with typical radii of 0.5 µm and heights of 60 nm are dispersed over the film’s surface. The grains’ mechanical properties are the same as those of the deposited film. The Berkovich indenter (R_c = 50 nm) was less than the grain radii for these measurements, therefore depth profiles of mechanical properties were observed for various types of contact between the tip and the selected grain. Finally, residual imprints were examined by AFM. The surface topography had a considerable impact on the near-surface mechanical properties, and determined hardness and elastic modulus were notably underestimated or overestimated in the range of 50% to 100% compared to the real values. Using cyclic nanoindentation on flat surfaces or the grain tops and extrapolating the depth profiles to the zero-contact depth is the proper solution.

9. “Performance evolution of inorganic fullerene nanoparticle lubrication additive using PI 95 in-situ TEM nanointender”

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The lubricating system used in internal combustion engines consists of oil and additives based formulations. Among the additives, anti-wear and friction modifying additives are considered very significant for the performance of the lubricant. Using nanoparticle as a potential additive proved to be an efficient alternative approach mainly because they are small enough to enter the contact area [1]. Recently inorganic fullerene-like – IF-based nanoparticles made up of metal disulphides MoS_2, WS_2 were developed and exhibit promising application potential. Their functional mechanism relies on the exfoliation of the external layer of the nanoparticle under mechanical stress leading to the formation of low friction tribofilms [2]. Optimizing the tribological property can be achieved by controlling the size, shape and chemistry of the IF nanoparticles since these parameters might have an impact on the exfoliation mechanism. A characterization by in-situ compression and/or friction inside transmission electron microscopy appears as a relevant tool to characterize the exfoliation mechanism. In this study, MoS_2 nanoparticles with controlled sizes were prepared by exposing the sheelites $MoMO_4$¬ nano particles to a H_2S-CCl_4-Ar gas mixture [3]. An isolated IF-MoS_2 nanoparticle was transferred to a sapphire substrate mounted on a copper pallet and the pallet is attached to Bruker’s Hysitron PI 95 in-situ TEM nanointender [4]. In-situ compression behavior of MoS_2 nanoparticles with different sizes were studied to observe in real time the morphological changes of nanoparticles under shear stress. These results were also related to friction experiments performed in a IF-MoS_2 lubricated contact under boundary lubrication regime.

[1] Spikes H., *Tribology Letters*, 60, 5 2015.

[2] Cizaire, L., et al., *Surface and Coatings Technology*, 160, 282, 2002.

[3] Afanasiev, P., *Appl. Catal. B: Environmental*, 227, 44 2018.

[4] R. Zhang, PhD thesis, INSA, University of Lyon, 2022.

Practical Information

Nanobrücken 2023

Nanobrücken 2023 will be held at Saarland University, located in the beautiful city of Saarbrücken.

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.

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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

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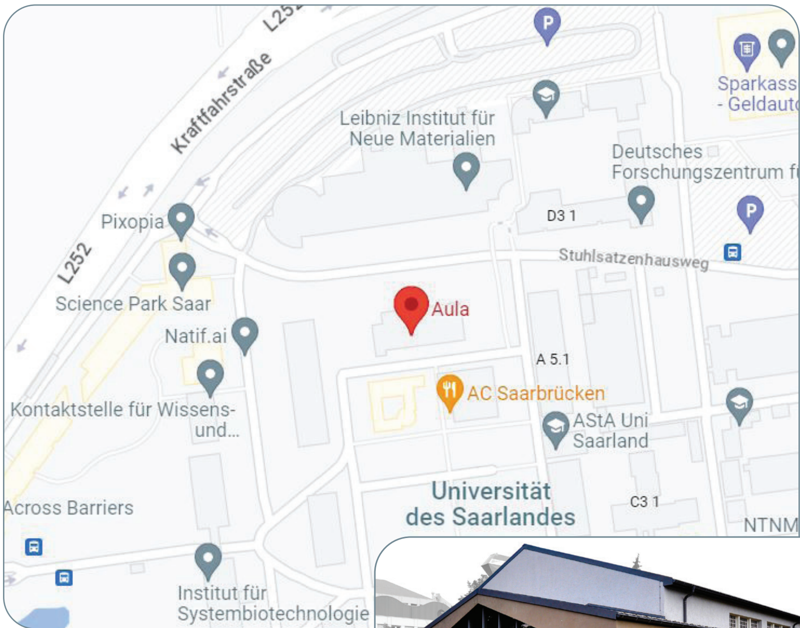

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