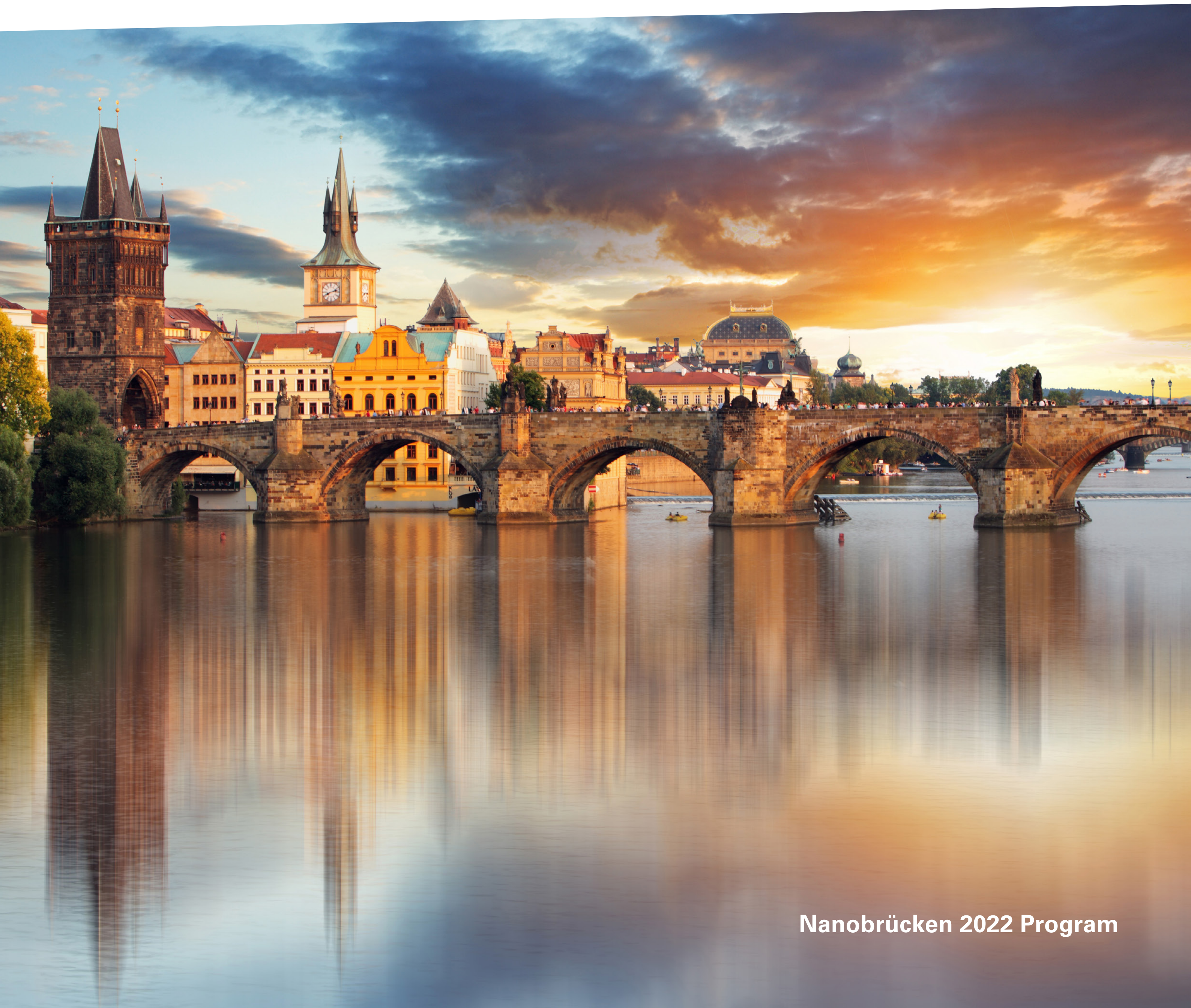
Nanobrücken 2022

A Nanomechanical Testing Conference
and Bruker User Meeting

June 8th - 10th, 2022 | Prague, Czech Republic



Nanobrücken 2022 Program

Preface

Bruker and Charles University are pleased to present **Nanobrücken 2022: Nanomechanical Testing Conference and Bruker User Meeting**, taking place June 8th-10th in the beautiful city of Prague.

This is the 12th 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

Charles University, Karolinum
Ovocny trh 5, 11636 Praha 1, Czech Republic

Program Committee

Conference Committee Members

Miroslav Cieslar, Charles University (Czech Republic)
Barbora Křivská, Charles University (Czech Republic)
Lucia Bajtošová, Charles University (Czech Republic)
Jaroslav Lukeš, Bruker (Czech Republic)
Ude Hangen, Bruker (Germany)

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Bruker Organizational Contacts

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

Day 1: Wednesday, June 8th

Welcome

13:30 Opening Remarks
Jaroslav Lukeš and Ude Hangen, Bruker

Student Talks — Session I

Chair: Ude Hangen, Bruker

13:45 "Small-scale mechanical testing of grain boundary doped ultrafine-grained W – Challenges and insights"
Michael Wurmshuber, Montanuniversität Leoben

14:00 "Properties of thin films studied by in-situ TEM"
Lucia Bajtošová, Charles University Prague

14:15 "Micromechanical testing approaches for local interface properties of interconnects in microelectronics"
Wieland Heyn, Fraunhofer IKTS

14:30 "Segregation-induced microscale strengthening of a Cu asymmetric Σ5 grain boundary doped with Ag"
Mohammed Kamran Bhat, MPIE Düsseldorf

14:45 "A quantitative analysis of stress-induced amorphization and grain boundary sliding in forsterite"
Ihtasham Ul Haq, University of Antwerp

15:00 "Mechanics of lithium metal at nanoscale by in-situ EBSD coupled nanoindentation"
Jack Aspinall, University of Oxford

Poster Set-Up

15:15 Short Break / Coffee Provided

Student Talks — Session II

Chair: Birte Riechers, BAM

15:45 "Microtensile Behavior of Additively Manufactured 17-4PH Stainless Steel Through In-Situ SEM Experimentation"
David Gonzalez-Nino, University Arkansas

16:00 "Nanoscale creep behavior and creep size effect of an additively manufactured Zr-based bulk metallic glass"
Siqi Liu, Norwegian University of Science and Technology

16:15 "Plastic instabilities in epitaxial NiMnGa Heusler films"
Adnan Fareed, BAM-Berlin

16:30 "Size effects in fracture mechanics: a detailed investigation on crack growth at the micro- and mesoscale"
Jutta Luksch, University of Saarbrücken

16:45 "Batch Fabrication of Silicon indenter tips for adhesion investigations using Deep Reactive Ion Etching (DRIE)"
Selina Raumel, Leibnitz University Hannover

17:00 "Following in-situ crack propagation"
Klemens Schmuck, Montanuniversität Leoben

Poster Session
All Posters are Eligible for Top Poster Prize

17:15 Poster Session (All posters, see list on page 7) / Barbecue

Bruker User Meeting (open to all participants)

19:00 Introduction
Oden Warren, Bruker

19:05 EDS for in-situ elemental analysis in SEM and TEM
Meiken Falke, Bruker

19:15 Exciting New Product Developments
Sanjit Bhowmick and Rhys Jones, Bruker

19:30 Round Table & Q&A Session (opportunity for users to ask questions)

20:00 End of User Meeting

Day 2: Thursday, June 9th

09:00 Nanobrücken Conference – Opening Remarks
Miroslav Cieslar, Charles University and Oden Warren, Bruker

Keynote Lecture
Chair: Miroslav Cieslar, Charles Uinversity Prague

09:10 **"Release with ease - challenges and bioinspired concepts for handling micro-objects"**
Prof. Eduard Arzt, Institute for New Materials, Saarbrücken

10:00 Short Break / Coffee Provided

Talks — Session I
Chair: Jaroslav Lukeš, Bruker

10:35 "Correlative Structure-Property Characterisation of the Leafcutter Ant, Atta cepholotes, Mandible"
Richard E. Johnston, Swansea University

10:55 "The cytolinker and scaffolding protein ''Plectin" disarray, leads to softening of cancer cells"
Anahid Amiri, TU Darmstadt

11:10 "Micromechanical Characterization of Enzyme Crystals"
Achim Overbeck, University of Braunschweig

Invited Talk
11:30 "Surfaces and Interfaces: Measuring Mechanical and Tribological Properties at the Nanoscale"
Jurgita Zekonyte, University of Portsmouth

Lunch Break
12:00 Lunch Provided On-Site

Talks — Session II
Chair: Sanjit Bhowmick, Bruker

13:00 "Combining electron microscopy and nanoindentation to characterize microstructural gradients in high-purity Niobium single crystals deformed at low and high strain rate"
Célia Caër, ENSTA Bretagne

13:20 “Recent developments on characterization of nanomaterials using On-Axis TKD in SEM”
Daniel Goran, Bruker

13:40 “Miniaturized mechanical tests in a laboratory X-ray microscope – Design, integration and applications”
Ehrenfried Zschech, deepXscan

14:00 “Developing rapid residual stress measurements via nanoindentation on a commercial aerospace alloy Al7050”
Elizabeth Sackett, Swansea University

Invited Talk

14:20 “Stress induced amorphization in olivine: new insights from ex-situ and in-situ TEM nanomechanical testing”
Hosni Idrissi, University Louvain La Neuve

14:50 Short Break / Coffee Provided

Talks — Session III

Chair: Jurgita Zekonyte, University of Portsmouth

15:20 “Measuring the mechanical damping using nano-compression tests”
Jose F. Gómez-Cortés, University of the Basque Country

15:40 “HCF fatigue testing and analysis of nanometer-sized copper bending beams”
Florian Schäfer, Universität des Saarlandes

16:00 “Micro-mechanical testing of highly air-sensitive Argyrodite by in-situ nanoindentation and micro-cantilever bending”
Johann Perera, University of Oxford

16:15 “Size effects at nanoscale on superelasticity measured by nano compression: Universal scaling law for Cu-Based shape memory alloys”
Jose M. San Juan, University of the Basque Country

16:35 “Elastic and Plastic Time Dependent Mechanical Properties of Lithium Measured by Nanoindentation”
J Ed Darnbrough, University of Oxford

16:55 End of Session

19:00 Conference Banquet Dinner

Day 3: Friday, June 10th

Talks — Session IV

Chair: Hosni Idrissi, Université Catholique de Louvain

Invited Talk

09:15 “Nanomechanical analysis of cement-based heterogeneous microstructures”
Jiri Nemecek, Czech Technical University in Prague

09:45 “In-situ nanoindentation of hard coatings at elevated temperatures”
Saeed Mirzaei, Academy of Science of the Czech Republic

10:05 “Mechanical properties of hard protective coatings for high temperature applications”
Aljaž Drnovšek, Jožef Stefan Institute

10:25 Short Break / Coffee Provided

Talks — Session V

Chair: Jose Maria San-Juan, University of the Basque Country

Invited Talk

10:50 "Nano-mechanical probing of elasticity length-scales in metallic glasses"
Birte Riechers, BAM

11:20 "Molecular Dynamics Simulations of Push-To-Pull Tests in Graphene Sheets"
Javier Varillas, Academy of Science of the Czech Republic

11:40 "Combining Nanoindentation and Simulation for elasto-plastic material data of thin metal films"
Nathaniel Jöhrmann, TU Chemnitz, Materials and Reliability of Microsystems

12:00 "Nanoindentation of thin films – accurate assessment of Young's modulus"
Stanislav Žák, Erich Schmidt Institute, Leoben

12:20 Closing Remarks and Farewell
Jaroslav Lukeš and Ude Hangen, Bruker

12:30 End of Conference

Poster List — Ordered by last name of presenting author

1. "Mechanical Properties of Interface Adhesion in Solid State Batteries"
   Shatha AlMarri, University of Oxford
2. "Nanoindentation and nanostructures flattening as a tool to obtain surface tension in chalcogenide glass-forming materials"
   Jaroslav Barták, University of Pardubice
3. "Nanomechanical study of PLA-based nanocomposite materials"
   Todor Batakliev, Institute of Mechanics, Bulgaria
4. "Advances in combined mechanical and electrical SPM characterization of thin films"
   Günther Benstetter, Deggendorf Institute of Technology
5. "Correlative Characterisation of Rangifer Tarandus (Reindeer) Antler, Evaluating Differences Between Male and Female and Calving Females"
   Rachel Board, Swansea University
6. "Local determination of stress-strain curves for multi-material semi-finished products made by laser build-up welding"
   Christoph Büdenbender, Leibniz Universität Hannover
7. "Comparison of methods for nanomechanical characterization of soft specimens"
   Daniel Haško, International Laser Centre, Slovak Republic
8. "Investigation of the adhesion of molybdenum and molybdenum trioxide to cube corner indenters by nanowear tests"
   Norman Heimes, Institute of Forming Technology and Machines, Leibniz Universität Hannover
9. "Effect of combination of carbon nanofillers on the nanomechanical properties of PLA-based composites films"
   Evgeni Ivanov, Institute of Mechanics, Sofia
10. "Different approaches for determination of the mechanical behavior changes in micro-structured glasses"
    Petr Knotek, University of Pardubice
11. "Local mechanical properties testing of zirconium alloy nuclear fuel claddings"
    Ondřej Libera, Centrum Výzkumu Řež
12. "Atomic Force Microscopy of Biosamples"
    Jan Přibyl, Masaryk University
13. "Surface properties of MXene sprayed films"
    Michal Procházka, Polymer institute, Bratislava
14. "Modified consolidants of porous materials with multifunctional performance"
    Monika Remzova, J. Heyrovsky Institute of Physical Chemistry of the CAS
15. "Development of a push to pull microsystem for tensile strength tests with cells of filamentous microorganism"
    Marcel Schrader, Technische Universität Braunschweig
16. "Micromechanical Properties of Native Human Ligamentum Flavum"
    Josef Sepitka, Czech Technical University in Prague
17. "Development of protocols for measuring twinning stress using micropillar compression"
    Camila A. Teixeira, IAM-KIT
18. "Near-surface viscosity in amorphous chalcogenides"
    Michaela Včeláková, University of Pardubice

Abstract - Oral Presentations

Day 1: Wednesday, June 8th

Student Talks — Session I

Chair: Ude Hangen, Bruker

13:45 "Small-scale mechanical testing of grain boundary doped ultrafine-grained W – Challenges and insights"

Michael Wurmshuber[1,*], Stefan Wurster[2], Severin Jakob[1], Markus Alfreider[1], Verena Maier-Kiener[1], Reinhard Pippan[2], Daniel Kiener[1]

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Grain refinement to the ultrafine-grained regime (grain sizes of 100-500 nm) is a popular method to improve the ductility and fracture toughness of W and render it feasible for employment in high-performance applications, such as nuclear fusion reactors. As both of these properties are ultimately limited by the grain boundary cohesion of W, doping with cohesion-enhancing elements yields a promising strategy to improve the mechanical properties even further. Given that ufg W today cannot be fabricated in large quantities, we have to rely on small-scale testing techniques to evaluate the effect of such grain boundary doping elements on the mechanical properties. As nanoindentation-based tensile tests in small dimensions are challenging and cannot be conveniently utilized for materials as hard and stiff as ufg W, relying on microcantilever bending tests seems most promising for the evaluation of (bending) strength and ductility. Regarding elastic-plastic fracture mechanical experiments, one has to be extremely careful that sample dimensions are large enough to fulfill the validity criterion, as fracture toughness can be both over- and underestimated depending on the ratio of plastic zone size to sample dimensions. This work presents the challenges that arise from small-scale testing of ufg W, while also investigating the effect of grain boundary doping on strength, ductility and fracture toughness. Thus, a pathway to designing more ductile nanoscale W alloys is created, while at the same time the application of small-scale testing techniques is defined, which will be useful for assessing future control samples employed during operation of a fusion reactor.

14:00 "In situ TEM and molecular dynamics study of tensile properties of nanocrystalline thin films"

Lucia Bajtošová[1,*], Barbora Křivská[1], Rostislav Králík[1], Jozef Veselý[1], Jan Hanuš[1], Petr Harcuba[1], Jan Fikar[2], Ankit Yadav[2], Miroslav Cieslar[1]

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The interest in the mechanical properties of thin films was evoked by their expanding application in micro- and nano-electro-mechanical systems such as thermal sensors or accelerometers. The dimensions of the thin films combined with restricted grain sizes in nanocrystalline films have an effect on their mechanical properties and deformation mechanisms. Tensile properties of Al-based nanocrystalline thin films were studied by in situ TEM. Deformation via grain rotations was confirmed by automated crystallographic orientation mapping (ACOM TEM). The straining of nanocrystalline thin films was then simulated by molecular dynamics to visualize the deformation mechanisms on the atomic scale. The results of the simulations were compatible with the experiment in the main features. In both cases, the films were deformed mainly by grain boundary mediated mechanisms while only restricted dislocation motion was observed. Intergranular failure of the films was confirmed.

14:15 "Micromechanical testing approaches for local interface properties of interconnects in microelectronics"

Wieland Heyn[1,3,*], Hanno Melzner[2], Klaus Goller[2], Sergey Ananiev[2], André Clausner[3], Johannes Zechner[2], Ehrenfried Zschech[1]

1. TU Dresden, Faculty of electrical and computer engineering, Germany
2. Infineon Technologies AG, Am Campeon 1-15, 85579 Neubiberg, Germany
3. Fraunhofer Institute for Ceramic Technologies and Systems IKTS, Maria-Reiche-Strasse 2, 01109 Dresden, Germany

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Adhesion testing methods for small wiring levels in microelectronic devices [back end-of-line (BEoL) levels], are critical for the manufacturing and reliability testing of modern microelectronic structures. However, only few measurement techniques exist, and moreover, they do not allow the inclusion of product identical size scaling or realistic structures in the study of interface properties. In this study, novel approaches are presented to perform direct mechanical tests on individual interconnect structures. For this, specially designed test structures are fabricated by Dual Damascene process. The test structures thereby exhibit product like properties. By using different geometries of test structures and by altering the experimental setup, it becomes possible to test the interfaces of interest in three loading modes. For loading Mode I a cantilever geometry is designed and tested in-situ in the SEM by using a PicoIndenter 87 tool. Furthermore, shear experiments are carried out, testing Cu pillars, using a 2D Transducer, equipped in a TI 950 nanoindentation system. The mentioned system is used to inflict torsional stresses on the third design, presented too. For the investigation of interface properties, the experimental load-displacement data is reviewed. FIB cross sections are performed to validate the delaminated interface. The results provide information on the critical delamination forces and show a good reproducibility. Furthermore, FEM simulations of the experiments are presented, aiming at evaluating the interface properties of the interfaces of interest i.e. interface strength and interface toughness.

14:30 "Segregation-induced microscale strengthening of a Cu asymmetric Σ5 grain boundary doped with Ag "

Mohammed Kamran Bhat[1,*], Lena Frommeyer[1], Prithiv T. Sukukumar[1], Gerhard Dehm[1], James P. Best[1]

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Grain boundaries (GBs) play a pivotal role and profoundly influence various properties such as electrical resistivity, mechanical strength or corrosion resistance, among others. While there have been notable attempts to explain grain-size strengthening, segregation induced embrittlement or GB slip transmission through both experiments and simulations a comprehensive understanding of the influence of various other parameters such as temperature, strain rate or local chemistry on dislocation-GB interactions does not yet exist, of paramount importance in predicting and controlling damage nucleation at these interfaces. In this work, the influence of GB chemistry on the mechanical response is studied through well-designed *in-situ* micropillar compression. GB chemistry is varied using Ag as a segregate species in Cu bicrystals with a penetrable Σ5 GB while limiting the size of the micropillars to 1 μm, ensuring dislocation-boundary interaction as the governing deformation mechanism. As Ag is almost immiscible in the Cu matrix, we analyse the mechanical responses of pure and Ag-doped GB to demonstrate the effect of Ag segregation on the deformation behaviour. Post deformation images combined with stress-strain data from micropillar compression tests indicate a possible contribution from local dislocation-solute interactions at the GB to the strength increase.

14:45 "A quantitative analysis of stress-induced amorphization and grain boundary sliding in forsterite"

Ihtasham Ul Haq[1,*], Patrick Cordier[2,3], Dominique Schryvers[1], Hosni Idrissi[4,1]

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2. Univ. Lille, Lille, France
3. Institut Universitaire de France, Paris, France
4. Université Catholique de Louvain, Louvain-la-Neuve, Belgium

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Olivine is the most abundant (> 60 % in volume) and weakest phase in the Earth's upper mantle of which it controls the rheology. This orthorhombic mineral does not have enough slip systems for homogeneous plastic strain (Von Mises criterion), other intergranular deformation mechanisms are thus needed to accommodate a general strain. Recently a stress-induced grain boundary sliding mechanism based on grain boundary amorphization was reported in olivine deformed experimentally [1]. Quantitative investigation of the elementary mechanisms involved in such a process are still missing. In this work, synthetic forsterite (the magnesium-rich endmember of the olivine solid-solution) samples are used. A PI-95 TEM Pico-indenter holder with a Push-to-Pull (PTP) device (Bruker. Inc) was used to perform quantitative tensile tests *in situ* in a transmission electron microscope (TEM). To observe grain boundary mechanisms, bi-crystals specimens were prepared from bulk pristine forsterite by focused ion beam (FIB). We show that the specimens deform exclusively by grain boundary sliding. Detailed microstructural investigation shows direct evidence of stress-induced amorphization in the sliding grain boundaries. Moreover, nanomechanical characterization of grain boundary sliding is achieved, providing for the first time, the constitutive equation describing the mechanical properties of a single grain boundary during amorphization and sliding. The elementary mechanisms at the origin of amorphization are investigated using high resolution TEM.

[1] Samae V. Cordier P., Demouchy S., Bollinger C., Gasc J., Koizumi S., Mussi A., Schryvers D. & Idrissi, H. "Stress-induced amorphization triggers deformation in the lithospheric mantle," *Nature*, vol. 591, 2021, doi: 10.1038/s41586-021-03238-3.

15:00 "Mechanics of lithium metal at nanoscale by in-situ EBSD coupled nanoindentation"

Jack Aspinall[1,*], J Ed Darnbrough[1], David EJ Armstrong[1], Mauro Pasta[1]

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The fracture of ceramic solid electrolytes, driven by the plating of lithium within submicron sized cracks, has been identified as one of the fundamental issues to resolve to successfully develop solid-state batteries[1]. Therefore a full understanding the mechanics of lithium at the nanoscale is essential. In this work, the elastic and plastic properties of lithium are measured by Berkovich in-situ nanoindentation within an enclosed argon glovebox system. Lithium is particularly challenging to study due to its high reactivity and softness, preventing the use of traditional surface preparation methods. Using a novel surface preparation methodology, lithium metal samples are characterised by electron backscattered diffraction (EBSD) before and after indentation to allow the dependence of the mechanical properties on crystallographic orientation to be measured, and the stiffness tensor components, moduli and poisons ratio to be independently determined using a method first proposed by Vlassak and Nix[2]. The measured stiffness tensor components are $C11 = 13.2$ GPa, $C12 = 11.1$ GPa and $C44 = 8.7$ GPa, giving an anisotropy ratio of 8.31 and a Poisson's ratio of 0.28. These values agree with earlier acoustic works on lithium single crystals within 3%[3]. Hardness measurements show anisotropy according to Schmid's law, with a clear size effect. The experiments are then repeated across lithium magnesium solid solution alloys from 5 to 20 atomic percent to investigate the change in anisotropy in elastic and plastic properties with solute content.

[1] E. Kazyak, R. Garcia-Mendez, W.S. LePage, A. Sharafi, A.L. Davis, A.J. Sanchez, K.H. Chen, C. Haslam, J. Sakamoto, N.P. Dasgupta, Li Penetration in Ceramic Solid Electrolytes: Operando Microscopy Analysis of Morphology, Propagation, and Reversibility, *Matter*. 2 (2020) 1025–1048. https://doi.org/10.1016/j.matt.2020.02.008.

[2] J.J. Vlassak, W.D. Nix, Measuring the Elastic Properties of Materials By Means of Indentation, *J. Mech. Phys. Solids*. 42 (1994) 1223–1245.

[3] T. Slotwinski, J. Trivisonno, Temperature dependence of the elastic constants of single crystal lithium, *J. Phys. Chem. Solids*. 30 (1969) 1276–1278. https://doi.org/10.1016/0022-3697(69)90386-2.

Student Talks — Session II

Chair: Birte Riechers, BAM

15:45 "Microtensile Behavior of Additively Manufactured 17-4PH Stainless Steel through In-Situ SEM Experimentation"

David Gonzalez-Nino[1], Gary S. Prinz[1,*]

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Metal additive manufacturing (AM) technologies such as selective laser melting (SLM) allow for rapid fabrication of geometrically complex parts that would be difficult to create using traditional casting or subtractive fabrication processes. In the SLM fabrication process, layers of fine metal powders are selectively melted together using controlled lasers to form three-dimensional part geometries. Research into AM metals has shown that fabrication defects resulting from SLM processes (i.e. voids, un-melted particles, etc.) can have deleterious effects on mechanical behavior. Material testing using traditional macro (coupon-scale) volumes may not accurately capture actual material behavior, as the distribution of fabrication defects is volume dependent. To understand fundamental material behavior at scales independent of geometrical fabrication defects, this project conducts in-situ SEM micro-tensile testing of AM SLM 17-4 PH stainless steel materials. To capture strain damage effects for later low-cycle fatigue inferences, the in-situ micro-tensile testing is performed on both unstrained, and pre-strained steel samples. Comparison between micro-tensile testing and bulk (macro) tensile testing indicate that the defect-free micro-tensile specimens have a greater ultimate tensile strength (1453MPa) and strain before failure (0.3741) than the macro-tensile specimens (1025 MPa tensile strength and 0.190 strain at fracture respectively). Micro-tensile material testing in AM metals allows for fundamental material property characterization and could allow for property upscaling to larger AM material volumes if geometric void distributions are known.

16:00 "Nanoscale creep behavior and creep size effect of an additively manufactured Zr-based bulk metallic glass"

Siqi Liu [1], Zhiliang Zhang [2], Jianying He[1,*]

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Nanoscale creep behaviors of metallic glasses (MGs) have gathered considerable interests in recent years, owing to their distinct atomistic mechanisms of plasticity. Additive manufacturing (AM) is a burgeoning technique for manufacturing MGs, the nanoscale creep characteristics and creep mechanism of the AM-ed MGs, however, remain ambiguous. In this work, the nanoscale creep behavior and creep size effect of a selective laser melted (SLM-ed) Zr-based MG were investigated by using nanoindentation. The creep stress exponent (n) and shear transformation zone (STZ) volume, as the indicators of the creep mechanism, were estimated. The n values are in the range of 2-3, and STZ volumes are around 2 nm3. The creep resistance of the MG was found to decrease with the increasing applied peak loads. A potential mechanism for this creep size effect was revealed: the smaller of the STZ volume, as well as the greater ratio of plastic flow under the higher maximum load, are responsible for the decreasing tendency of creep resistance. This research gives a comprehensive understanding of the atomistic mechanisms in the AM-ed MG during the nanoscale creep deformation process and can serve as a reference for improving the plasticity and further engineering applications.

16:15 "Plastic instabilities in epitaxial NiMnGa Heusler films"

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Magnetic shape memory alloys are known to undergo stress- and temperature-driven phase changes. Here we study the specific case of a NiMnGa Heusler alloy that has an austenitic phase at room temperature. Upon cooling or the application of mechanical pressure, the austenite can transform into martensite, allowing for large reversible strain cycling and making such alloys to promising actuating materials. In order to shed more light on the mechanical switching behavior and possible dissipative processes, we probe the nano-scale plasticity of 0.5 and 2 µm thick epitaxial NiMnGa films with nanoindentation. A distinct pop-in signature is seen as the first departure from Hertzian elastic contact mechanics at small film thicknesses. This pop-in behavior persists across four orders of loading rates and over a broad temperature regime from 40°C to -30°C, which encompasses the transformation temperature to martensite. The statistics of the incipient plastic events are well described by a Weibull distribution. Atomic force microscopy reveals surface signatures around indents that indicate residual martensite, which is further confirmed with transmission electron microscopy imaging of the structure underneath indents. Instead of the expected modulated martensite (14M, 10M) that forms during a temperature-driven phase change, regions underneath indents contain non-modulated (NM) martensite. NM martensite exhibits a higher spontaneous strain and often forms at lower temperatures and higher strains. Therefore, it is concluded that the pop-in signature during nanoindentation originates from an athermal martensitic transformation, where the confinement effects result in huge and complex deformation inducing a partly irreversible transition to NM martensite.

16:30 "Size effects in fracture mechanics: a detailed investigation on crack growth at the micro- and mesoscale"

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

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Fracture mechanics are strictly regulated by standards. This includes limitations of specimen size as a function of plastic zone size. Since the size of the plastic zone mainly depends on the material itself, the established macroscopic test procedures cannot be easily downscaled when sample size decreases to the microscale. With advances in micromechanical testing, the classical size effect in metals, whereby the strength increases as specimen dimensions become smaller, has been studied in detail. However, potential size effects in fracture mechanics, which are important for designing components and predicting their lifetimes, has not been adequately explored. With progress in specimen preparation using focused ion beam and designing the mechanical tests, for e.g. in-situ testing inside scanning electron microscope, it is possible to investigate this size effect systematically. In the present study, nanocrystalline nickel, with nominal mean grain size of 40-50 nm, was used as a model material to ensure a polycrystalline, quasi-homogeneous microstructure even for small samples. Micro-bending beams of cross-sectional area of a few tens of μm^2 up to a few thousands of μm^2 were fabricated using a focused ion beam in order to study size effects in fatigue testing. Special attention was given to introduce a fatigue pre-crack in the sample by cyclic loading with $R<0$ using an in-situ nanoindenter inside a scanning electron microscope. This crack was then subjected to fatigue test ($R>0$) and the crack growth was quantified by the compliance method. In addition, the stress intensity factor was evaluated and related to the crack growth.

16:45 "Batch Fabrication of Silicon indenter tips for adhesion investigations using Deep Reactive Ion Etching (DRIE)"

Selina Raumel[1,*], Folke Dencker[1], Marc Christopher Wurz[1,2]

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2. DLR Institute of Quantum Technologies, Ulm University, Germany

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To investigate the adhesion tendency of two parallel surfaces without interfering macroscopic and microscopic effects, functionalized flat indentation tips with minimal surface area and metallization were produced in a photolithographic batch process.

For the fabrication of the cylindrical silicon flat-tips, columns with different diameters between 8 and 50 µm and heights between 60 and 80 µm were fabricated by deep reactive ion etching (DRIE) from silicon wafers using a photoresist mask patterned by standard lithography. The mask designs included stand-alone tips with different geometries as well as tips with additional sacrificial structures to improve the etching process. Suitable process parameters were determined to achieve a sufficient edge accuracy with an angle of at least 88°. The processed columns were separated into 600 x 600 µm² chips by a dicing process. After dicing, various approaches were investigated to join the fabricated columns on a milled rotationally symmetric shaft made of aluminum. The mechanical strength of the joining zone was then analyzed using shear tests and resulted in an average shear strength of 4.4 ± 0.15 N/mm².

The geometry, edge accuracy and plane parallelism of the joint were examined using SEM. Subsequently, the tips were metallized by cathode sputtering in order to investigate different material combinations. For characterization, the tips were installed in the Hysitron Tribondenter TI 900 to generate specific surface pressures between the surfaces of the tip and the workpiece in different atmospheres with and without relative motion, and then qualitatively investigated using SEM and EDS.

17:00 "Following in-situ crack propagation"

Klemens Schmuck[1,*], Martin Antenreiter[2], Daniel Kiener[1]

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Ongoing device miniaturization demands fracture mechanical investigations on ever smaller length scales. Accurate knowledge about the crack propagation is crucial to fracture experiments on all length scales. The crack propagation at the micron-to submicron scale is accessible indirectly via continuous stiffness measurements and/or directly by image based observations. For the latter, crack propagation is usually evaluated manually which is a tedious user-dependent task. Therefore, we developed a semi-automatic method based on probability-based segmentation of crack propagation. For verification, fracture experiments were performed with notched micro cantilever beams, which were fabricated from an ultra-fine grained W-Cu composite by focused ion beam milling. The cantilevers were tested by quasi-static loading using an ASMEC micro indenter, situated within a scanning electron microscope to record in-situ images. Obtained crack lengths by the semi-automatic method agree well with the manual assessment as well as with determined crack lengths from unloading stiffness. Further, the image based method accelerates evaluation compared to manual measurements. Thus, more data can be analyzed, which allows to investigate the fracture process in more detail. Besides that, the proposed method is less user-dependent and may allow to gain further fracture characteristics such as crack tip opening angle and crack tip opening displacement, which would enhance the fracture characterization further.

Abstract - Oral Presentations

Day 2: Thursday, June 9th

Keynote Lecture

Chair: Miroslav Cieslar, Charles Uinversity Prague

09:10 "Release with ease - challenges and bioinspired concepts for handling micro-objects

Prof. Eduard Arzt[1]

1. INM – Leibniz Institute for New Materials and Saarland University, Saarbrücken, Germany

Contact mechanics is strongly size dependent: In small dimensions, objects tend to tenaciously stick to each other. This poses severe challenges for microtechnology and the assembly of micro-components. Inspired by biological evolution, fibrillar surface microstructures have become an innovative and sustainable strategy for robotic gripping and handling. Following extensive development, this new technology is now on the path to commercialization. However, as the components to be handled are shrinking in size, their weight will be too small to allow detachment and placement by conventional mechanisms. Our group has proposed various designs that enable controlled manipulation of micro-objects. With an unprecedented switching ratio (between high and low adhesion) exceeding 50, such microstructure concepts may enable an energy-efficient solution to handling problems under extreme conditions.

Talks — Session I

Chair: Jaroslav Lukeš, Bruker

10:35 "Correlative Structure-Property Characterisation of the Leafcutter Ant, Atta cepholotes, Mandible"

Richard E Johnston[1,*], Mohammad Wakib Said[1], David Labonte[2], James Russell1, Elizabeth Sackett[1], Rachel Board[1]

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Materials formed in nature, often driven by evolution, perform challenging mechanical functions. This study looks at leafcutter ants, *Atta cephalotes*. The mandibles of the leafcutter ant, with zinc-tipped teeth, are used as cutting tools. In this study, multiple correlated characterisation techniques are connected to analyse the surface and sub-surface layers within the zinc-rich region of the *Atta cepholotes* mandible. Initial X-ray microscopy/microtomography (XRM/μCT) served as a 'bioprospecting' tool, identifying discrete layering in the zinc-rich outer region, warranting further investigation of possible structure-property/form-function relationships. Optical, SEM/EDS were carried out to identify the microstructure and map the elemental composition. Nanoindentation was carried out on a Bruker Hysitron Ti950 using a cube corner tip geometry, 800 μN load at 5 μm spacing, generating XPM hardness and reduced Modulus maps of the outer and intermediate layers of the mandibular teeth, and the cuticle beneath. The optical, SEM, EDS, and nanoindentation data was aligned to the specific plane within the original XRM dataset to provide a correlative multimodal view of the same regions. The Zn content correlated to the nanoindentation XPM maps, with higher zinc content in the outer layer providing greater hardness and Modulus, lowest in the surrounding cuticle, with intermediate mechanical properties for the intermediate layer. The combination of techniques in a correlative workflow enables us to probe the potential for materials science and biology crossover. Understanding structure-property relationships can identify possible form and function in nature. Learning from this structure could inform new material design for challenging applications.

10:55 "The cytolinker and scaffolding protein ''Plectin" disarray, leads to softening of cancer cells"

Anahid Amiri[1,*], Christian Dietz[1], Robert W. Stark[1]

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We started a research on the reason underlying the softening of cancerous cells in comparison to their counterparts based on Atomic Force Microscopy (AFM) with high special and depth precision in concert with Fluorescence Microscopy (FM). In our previous studies we have shown several cancer cell lines which present cytoskeletal distortion while showing lower bending rigidity compared to the healthy counterparts [1] or well differentiated cancer cells [2, 3]. The integrated level of both actin and tubulin moieties showed comparably lower levels in highly invasive cells. Despite the fact that the cytoskeleton components are generally decreased in amount and they form abnormally which can justify the softening of these cells, still the present ones in the cells, should resist the applied force and be sensed by the probe in force spectroscopy. Surprisingly, this expectation was not the observed scenario on cancer cells in our studies at which the AFM images were showing approximately no support of cytoskeleton on top of cells' nuclei. Furthermore, we observed MTs undergoing bending at the periphery of ductal carcinomas in an uncontrolled continuous manner and based on AFM force volume mapping images combined with Fluorescence imaging, MTs are situated around approximately 400 nm lower to the plasma membrane (cancer cell's cross-sectional profile (x-z) of the elastic modulus) while in normal cells the belt of MTs situated directly under the blend of F-actin and plasma membrane (I. normal cell's cross-sectional profile (x-z) of the elastic modulus). These observations drove our interest further to look into the cytoskeleton component joints (Plectin protein) of the cancer cells. These huge sized proteins (>500 kDa) cross-links intermediate filaments (IFs) to MTs and F-actin and mediates the attachment of IFs to membrane components. Plectin protein in normal cells localized in the expected peripheral areas of cells while these proteins are centralized on top of the nucleus in cancer cells, and are mis-localized to the cell surface. It stands to reason, in contrast to the presence of these proteins in the cancer cells, these proteins do not fulfill the task of crisscrossing IF and MTs, to maintain the tubules right beneath the plasma membrane and cell cortex blend.

[1] Amiri, A., et al., Structural analysis of healthy and cancerous epithelial-type breast cells by nanomechanical spectroscopy allows us to obtain peculiarities of the skeleton and junctions. *Nanoscale Advances*, 2019. **1**(12): p. 4853-4862.

[2] A. Amiri, F.D.H., C. Dietz, Carcinomas with Occult Metastasis Potential: Diagnosis/Prognosis Accuracy Improvement by Means of Force Spectroscopy. *Advanced Biosystems*, 2020. **4**(7): p. 2000042.

[3] Amiri, A., et al., Reliability of cancer cell elasticity in force microscopy. *Applied Physics Letters*, 2020. **116**(8).

11:10 "Micromechanical Characterization of Enzyme Crystals"

Achim Overbeck[1,2,*], Marta Kubiak[1,2], Benjamin Radel[3], Hermann Nirschl[3], Ingo Kampen[1,2], Arno Kwade[1,2]

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The industrial use of biocatalysts, such as isolated enzymes, is often limited by the lack of mechanical stability and reusability. To counteract this, free enzymes can be crystallized and immobilized. Because the processability and catalytic activity of such particles are influenced by the particle size, sufficiently mechanical stability is required to avoid crystal breakage, during e.q. a filtration step.

In order to simulate a load on rod-like crystals as close as possible to the loads occurring in the process, a three-point bending test was established using the TriboIndenter. Together with the structure of the filter cake determined by micro X-ray tomography, the data is available for a digital twin of filtration experiments.

Furthermore, nanoindentation experiments were carried out on native and cross-linked crystals. These measurements show a clear anisotropy of the crystals regarding their mechanical properties. The increase in crystal stability due to cross-linking could also be demonstrated with the smallest sample quantities.

Invited Talk

11:30 "Surfaces and Interfaces: Measuring Mechanical and Tribological Properties at the Nanoscale"

Jurgita Zekonyte[1,*]

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Since the development of nanomechanical testing techniques, it was possible to measure not only hardness but also elasticity, creep, as well as friction and wear and many more. As nanoscale indents can be targeted in localized areas as well as near surface region, the material properties extracted from those measurements are most relevant to understanding surfaces and interfaces of various materials for wide range applications including nanotribology. This presentation will cover few studies of different materials, ranging from biomedical to pure engineering applications, and how the nanoindentation results compliment and predict tribological response, i.e. friction, wear, erosion.

Talks — Session II

Chair: Sanjit Bhowmick, Bruker

13:00 "Combining electron microscopy and nanoindentation to characterize microstructural gradients in high-purity Niobium single crystals deformed at low and high strain rate"

C. Caër[1,*], J.F. Croteau[1,2], E. Cantergliani[2], N. Jacques[1]

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Manufacturing of bulk Niobium superconducting radiofrequency (SRF) cavities with improved performance and reduced cost is paramount for enabling future particle accelerators with increased collision energy [1]. An innovative option would be to use large grain-niobium manufactured using electro-hydraulic forming (EHF), a high velocity forming technique. These process changes may be beneficial for cavity performance and cost reduction, but require a detailed understanding of the high-strain rate forming process consequences on the materials microstructure.

This study investigates high-purity Niobium single crystals from which several specimen were extracted according to different crystallographic orientations before being mechanically loaded either at low or high strain rate until failure of the specimen [2]. EBSD maps and nanoindentation were used to characterize and correlate microstructural and mechanical gradients all along the samples gauge length from the grip to the fracture surface. It is shown that both crystal local reorientation and micro-plasticity are greatly affected by the prescribed strain-rate and the initial sample crystallographic orientation.

Acknowledgments:

EASITrain – European Advanced Superconductivity Innovation and Training. This Marie Sklodowska-Curie Action (MSCA) Innovative Training Networks (ITN) has received funding from the European Union's H2020 Framework Programme under Grant Agreement no. 764879.

[1] P. Kneisel et al., Preliminary results from single crystal and very large crystal niobium cavities, *Proceedings of the 2005 Particules Accelerator Conference* (2005) 3991-3993.

[2] J.-F. Croteau et al., Effects of strain rate on tensile mechanical properties of high-purity niobium single crystals for SRF applications, *Materials Science & Engineering A* **797** (2020).

13:20 "Recent developments on characterization of nanomaterials using On-Axis TKD in SEM"

Daniel Goran[1,*]

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Transmission Kikuchi Diffraction (TKD) in a Scanning Electron Microscope (SEM) has been first proposed in 2012 and has quickly become an established technique due to its much better spatial resolution as 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 improvements in signal yield and reduce gnomonic distortions in the patterns. These have led to significant gains in spatial resolution, data acquisition speed and data integrity. These gains 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 hardware and software developments intended to push even further the spatial resolution limit and data quality. Efforts to automate, time resolved mapping and imaging of nanomaterials and thin films during dynamic experiments, e.g. in-situ heating and/or electrical biasing, will be part of the discussion. 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.

13:40 "Miniaturized mechanical tests in a laboratory X-ray microscope – Design, integration and applications"

Ehrenfried Zschech[1,*], Kristina Kutukova[1], Martyna Strag[2]

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The combination of micromechanical testing and high-resolution X-ray imaging provides a better understanding of the fracture mechanics at small scales and it opens the way for the development of design concepts for novel engineered materials systems based on their local mechanical properties. Miniaturized mechanical tests for 3D-structured systems and materials, integrated in a laboratory X-ray microscope, represent a unique capability for sub-100nm resolution 3D imaging of microcrack evolution while a force is applied. Specially designed experimental setups are needed to grow microcracks in usually hierarchically structured material systems by applying a precisely controlled mechanical load and by monitoring force and displacements in materials at the micro- and nanoscale. Two applications of nano-XCT for imaging of microcrack evolution will be demonstrated: in a natural biocomposite and in a microchip. The 3D visualization of crack propagation in the protecting outer layer of a mollusk shell was performed using a customized micro-indentation system that was integrated in the nano-XCT system in such a way that it fits into the limited space of the tool and that the indenter tip and the sample fit into the field of view of the X-ray microscope. This study revealed that firstly microcracks propagate along interfaces with low resistance against crack growth, and subsequently these microcracks are steered to regions where they are stopped. The 3D visualization of crack propagation in advanced integrated circuits was performed using a micro double cantilever beam test in an X-ray microscope. It was possible to control fracture and to steer cracks to regions in the on-chip interconnect stack with relatively high fracture toughness.

14:00 "Developing rapid residual stress measurements via nanoindentation on a commercial aerospace alloy Al7050"

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Measuring residual stresses in aerospace components reliably in a fast, cost-effective manner presents a significant challenge. Therefore, developing a rapid, robust technique using nanoindentation would be beneficial to address industrial requirements, and would allow measurements at the point of manufacture without the need for X-rays and the associated safety mitigations necessary. To assess the suitability of using nanoindentation as a rapid residual stress measurement technique, a comparative study will be carried out to compare the results from nanoindentation to samples that have been characterized via high confidence diffraction methods. Using an array of quenched blocks of Al7050 produced with a range of quenching parameters and with additional heat treatments to provide a wide range of residual stresses. A blind study using coded blocks will be presented on results from the nano indenter that had been previously characterized using diffraction techniques. This investigation will also assess the sensitivity of residual stress measurements using nanoindentation on Al7050.

Invited Talk

14:20 "Stress induced amorphization in olivine: new insights from ex-situ and in-situ TEM nanomechanical testing"

Hosni Idrissi[1,2,*] and Patrick Cordier[3,4]

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3. Univ. Lille, CNRS, INRAE, ENSCL, UMR 8207 - UMET - Unité Matériaux et Transformations, F-59000 Lille, France
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This work is motivated by the recent discovery of a new deformation mechanism responsible for grain boundary sliding in olivine: stress induced amorphization [1]. This mechanism operating under high stress could play an important role in geodynamics to account for the ductile brittle limit (responsible for earthquakes) and the major rheological transition between the lithosphere and the asthenosphere. Based on our expertise in nanomechanical testing and transmission electron microscopy (TEM), we propose an original approach to understand and quantify the elementary microscopic mechanisms responsible for this phenomenon in order to model the mechanical behaviour of rocks under these conditions. We have performed in situ deformation tests in TEM to study the activation of amorphization at grain boundaries. The intergranular amorphous phase is then isolated to study its rheological properties thanks to a novel combination of tests with complementary capabilities: nanoindentation, in situ TEM deformation and Lab-on-Chip. These tests have in particular the capacity to extend the solicitations to very low deformation rates relevant in geodynamics. Advanced TEM characterizations have allowed to identify the underlying microscopic mechanisms either in-situ or in ex-situ deformed samples. These data will allow in the future to feed mesoscopic mechanical models of olivine-rich rocks of the upper mantle.

[1] V. Samae, P. Cordier, S. Demouchy, C. Bollinger, J. Gasc, S. Koizumi, A. Mussi, D. Schryvers, H. Idrissi, Stressinduced amorphization triggers deformation in the lithospheric mantle, *Nature*, 591, 2021, 82.

Talks — Session III

Chair: Jurgita Zekonyte, University of Portsmouth

15:20 "Measuring the mechanical damping using nano-compression tests"

Jose F. Gómez-Cortés[1,*], María L. Nó[1], Jose M. San Juan[1]

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Mechanical damping is the energy attenuation of a mechanical force disturbance, and this property is distinctive in Shape Memory Alloys (SMA) thanks to its superelastic behaviour (SB). The SB results from a stress-induced martensitic transformation on an austenite phase that is completely reversible when the stress is released. The output signal of one SB cycle draws a hysteresis loop on the stress-strain field, and this loop area offers a quantitative way to index the energy dissipation per cycle, using the loss factor η, which is an index ratio between dissipated energy (hysteresis cycle area) and the total applied energy (load area). The mechanical damping capacity linked to this energy dissipation was studied in Cu-Al-Ni SMA at the nano-scale using nano-compression tests on micro-pillars, getting outstanding results in terms of a superlative damping capacity (ultrahigh damping $\eta>0.2$) and long-term reliability (>5000 cycles) [1]. Figure 1 (not shown) depicts a superelastic response of a micropillar under a nano-compression test, framed in both fields, stress-train and load-displacement. In this work, we use a Hysitron IT 950 equipment to perform nano-compression tests, study the superelastic damping capacity in diverse Cu-based SMA, and compare loss factor results at the nano-scale with previous findings in the Cu-Al-Ni SMA [2].

[1] J.F. Gómez-Cortés, et al, *Acta Mater,* **166** (2019) 346-356.

[2] J.F. Gómez-Cortes, et al, *J. Alloys Compd.*, **883** (2021) 160865.

15:40 "HCF fatigue testing and analysis of nanometer-sized copper bending beams"

Florian Schaefer[1,*], Lukas Schon[1], Jorge Rafael Velayarce[1], Christian Motz[1]

1. Materials Science and Methods, Saarland University, Saarbruecken, Germany

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As long as the samples are large compared to the atomistic scale, the material behavior can be described as a continuum, especially in fatigue applications. The dislocation arrangements that form during the fatigue of face-centered cubic materials are well-known and typically have structural dimensions ranging from a few hundred nanometers to a few micrometers. But what happens when the sample thickness of a bending specimen is less than one micrometer? To answer this question, micro-cantilevers prepared in a focused ion beam were cyclically loaded in a Dimension Icon atomic force microscope up to several million load cycles. The large stress gradients halve the effective beam thickness. However, a classical Woehler behavior was still observed. The dislocation networks formed no longer correspond to the fatigue structures known from macro-sized samples. Moreover, the specimen thickness also determines the fatigue life for nanometer-sized specimens as known from thin film fatigue testing.

16:00 "Micro-mechanical testing of highly air-sensitive Argyrodite by in-situ nanoindentation and micro-cantilever bending"

Johann Perera[1,*], J Ed Darnbrough[1], Peter G Bruce[1,2], David EJ Armstrong[1]

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All-solid-state-batteries (ASSBs) pave the way for safe use of lithium-metal electrodes due to their use of a solid electrolyte (SE). Short circuit failure caused by penetration lithium dendrites through SEs remains one of the largest challenges facing ASSBs [1], it is therefore essential to gain a better understanding of the mechanical properties of SEs. Due to the highly air-sensitive nature of SEs conventional mechanical testing techniques are not possible and as a result very little is known about their mechanical properties [2]. In this work, the mechanical properties of Argyrodite, an air-sensitive sulphide, was investigated in an enclosed Argon glovebox system using in-situ nanoindentation. Both Berkovich and cube-corner nanoindentation testing procedures were used to measure elastic, plastic and fracture properties. A modulus of 28.0 GPa ± 1.8, hardness of 921 MPa ± 196 and a fracture toughness of 0.69 MPa $m^{1/2}$ ± 0.12 was measured. It has been shown that lithium dendrites are able to plated intergranularly through SEs, along the grain boundaries, leading to transgranular fracture of the grains [3]. This leads to the question was to what is the grain boundary strength of these SEs. Using an innovative technique, the grain boundary strength was measured to be 91.1 MPa ± 14.5. This was achieved by preparing a pentagonal microcantilever containing a grain boundary at its fixed end and by bending the free end using cube-corner nanoindentation. This value demonstrates the pressure which the Li metal must exceed to be able to ingress into the SE.

[1] Famprikis, T., Canepa, P., Dawson, J. A., Saiful Islam, M. & Masquelier, C. Fundamentals of inorganic solid-state electrolytes for batteries. doi:10.1038/s41563-019-0431-3.

[2] McGrogan, F. P. et al. Compliant Yet Brittle Mechanical Behavior of Li2S–P2S5 Lithium-Ion-Conducting Solid Electrolyte. *Advanced Energy Materials* **7**, 1602011 (2017).

[3] Cheng, E. J., Sharafi, A. & Sakamoto, J. Intergranular Li metal propagation through polycrystalline Li6.25Al0.25La3Zr2O12 ceramic electrolyte. *Electrochemica Acta* 85–91 (2017).

16:15 "Size effects at nanoscale on superelasticity measured by nano compression: Universal scaling law for Cu-Based shape memory alloys"

Jose M. San Juan[1,*], Jose F. Gómez-Cortés[1], Maria L. Nó[1]

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In recent years, an increasing interest is being devoted to the study of the size-effects on plasticity and phase transformations. In the case of shape memory alloys (SMA), these size-effects on the thermal or stress-induced martensitic transformation (MT) are relevant for the design of micro/nano actuators based on SMA. The Hysitron Nanoindenter TI-950 and the Picoindenter PI-85 were used to study the size effects on the superelastic behavior of micro and nano pillars milled by Focused Ion Beam in a FEI Helios Nanolab 650. Several series of pillars, from 2 micrometers down to 250 nm were milled in different Cu-based SMA [1,2]. The results allow establish a universal scaling law for the critical stress during the superelastic behavior. The present talk will offer an overview of the methodology for such experiments and on the observed size effects, which will be discussed in terms of the elastic and atomic models.

[1] J.F. Gómez-Cortés et al., *Nature Nanotechnology*, **12** (2017) 790.

[2] V. Fuster, et al., *Advanced Electronic Materials,* **6** (2020) 1900741.

16:35 "Elastic and Plastic Time Dependent Mechanical Properties of Lithium measured by nanoindentation"

J Ed Darnbrough[1,*], Jack Aspinall[1], Mauro Pasta[1], David EJ Armstrong[1]

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Metallic Lithium is the ultimate anode material for the highest energy density solid state batteries. However, the basic mechanical prop ties reported in the literature are inconsistent. An improved understanding of the basic mechanical properties of lithium is key for developing better solid state batteries. In this paper we use in-situ nanoindentation to measure the effect of strain rate and temperature on hardness that has an impact on battery cycle performance. This work clarifies the reasons for the range of property values reported in the literature with a global equation for yield strength. We show the pressure dependence of lithium for a given strain-rate can be changed by up to two orders of magnitude by the pre-existing dislocation structure and the recoverable and non-recoverable stress, illustrating that it is crucial to consider the materials history when defining the creep properties. These findings are in agreement with the extremes illustrated in the literature by the small scale pillars of Xu et al, where the size of the volume tested reduces the absolute number of dislocations, and by the delayed tests of Masias et al where the material relaxes reducing the amount of plastic strain and mobile dislocations leading to lower strain-rates at the same pressure. The dislocation density in Li is easy to change due to the low yield stress but the dislocation's permanence for aiding cycling could be effected by the ease of stress relaxation. Further study is required to consider if the athermal defects, which are unaffected by thermal relaxation, are able to enhance the creep properties.

[1] C. Xu, Z. Ahmad, A. Aryanfar, V. Viswanathan, and J. R. Greer. Enhanced strength and temperature dependence of mechanical properties of Li at small scales and its implications for Li metal anodes. *Proc. Natl. Acad. Sci.*, **114**(1):57{61, jan 2017. ISSN 0027-8424. doi: 10.1073/PNAS.1615733114.

[2] A. Masias, N. Felten, and J. Sakamoto. Characterizing the mechanical behavior of lithium in compression. *J. Mater. Res.* 2021 363, **36**(3):729{739, jan 2021. ISSN 2044-5326. doi: 10.1557/S43578-020-00028-X.

Abstract - Oral Presentations

Day 3: Friday, June 10th

Talks — Session IV

Chair: Hosni Idrissi, Université Catholique de Louvain

Invited Talk

09:15 "Nanomechanical analysis of cement-based heterogeneous microstructures"

Jiří Němeček[1,*], Radim Čtvrtlík[2], V. Šmilauer[1]

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Nanoindentation in its various modes is often used for characterization of homogenous-like bulk or thin film materials. In contrast, cement-based materials are posing a high degree of heterogeneity at all length scales of the material starting from nanometer to meter level. The contribution presents an overview of characterization of the material at nano- to micrometer levels by nanoindentation collected by the authors over a decade. The overview starts with the evaluation of elastic properties and hardness of several microstructural phases using statistical deconvolution technique. Then it presents evaluation of time dependent creep characteristics and fracture properties derived from crack monitoring and energetic methods, measurements of tensile strength and fracture energy on FIB prepared micro-beams and utilization of surface scratching accompanied by SEM and acoustic emission analyses.

09:45 “In-situ nanoindentation of hard coatings at elevated temperatures”

Saeed Mirzaei[1,2,*], Vilma Bursikova[3]

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The machining industry is always imperatively looking for increased cutting velocity and feed rates to provide cost-effective and long-lasting products. Therefore, coatings with outstanding properties, among which thermal stability is of great interest, are required for the cutting tool application. During the cutting process, the workpiece in the contact area heats up to temperatures up to 1000°C temperature. There are several industrial hard coatings in the market, such as TiAlN, CrAlN, or TiB_2, which exhibit good mechanical properties, but on the other hand, high brittleness, especial at elevated temperatures. It is a well-established concept that lack of ductility due to rapid crack propagation can lead to a premature failure of the coating and, consequently, a reduced lifetime of the cutting. Therefore, developing a hard coating with improved ductility, especially at elevated temperatures, is necessary. Nanocomposite hard coatings are an example of widely studied materials for this purpose. In this work, a Hysitron TI 950 instrument equipped with an xSol 800 stage has been used to evaluate the mechanical properties of nanocomposite coatings synthesized by a pulsed DC magnetron sputtering system. The hardness and reduced elastic modulus of the coatings have been measured both at room temperature and elevated temperatures. The xSol stage was able to actively heat the sample to ~800°C, however, the maximum temperature that the coatings could withstand was up to 600°C. High-temperature in-situ nanoindentation results were compared to the results of ex-situ measurements.

10:05 “Mechanical properties of hard protective coatings for high temperature applications”

Aljaž Drnovšek[1,*], Hi Vo[2], Marisa Rebelo de Figueiredo[3], Shraddha J. Vachhani[4], Peter Hosemann[2], Robert Franz[3] and Miha Čekada[1]

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Mechanical properties of protective coatings are commonly determined by nanoindentation methods. The development of nanoindentation in recent years led to new ex-situ and in-situ systems which are capable of measuring mechanical properties such as hardness, elastic modulus and fracture toughness at high temperatures too. For hard protective coatings it is of paramount value to gain knowledge of their mechanical properties close to the real operation temperature. We present the hardness and fracture toughness measurements at room temperature as well as at high temperature of typical nitride based hard coatings used for cutting applications, all deposited in an industrial deposition unit. For the determination of hardness, the stand alone nanoindentation system equipped with a hot stage was used, while the fracture toughness was determined by in-situ cantilever bending tests. Challenges in testing mechanical properties of thin protective coatings are discussed. The intention of this data is to serve as a first step towards a more comprehensive understanding of the mechanical properties of hard coatings that are used in HT applications.

Chair: Jose Maria San-Juan, University of the Basque Country

Invited Talk

10:50 "Nano-mechanical probing of elasticity length-scales in metallic glasses"

B. Riechers[1,*], R. Maass[1,2,*]

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Metallic glasses have a disordered atomic structure beyond some short- or medium-range order that is typically associated with either nearest neighbor atoms or the connectivity of a few polyhedra. Indeed, the ability of forming interconnected clusters of, for example, icosahedral structures has been demonstrated in atomistic simulations, giving direct insight into emerging structural length scales (*JALCOM* **821**, 153209, 2020). At the experimental scale one therefore expects to quickly average over the underlying structural heterogeneities and a uniform signal from the probed volume element should be obtained. A central question in the field has been at which length scale averaging is sufficient. As discussed in an earlier work (*Adv. Func. Mat.* **28**, 1800388, 2018), determining experimentally accessible length scales that possibly represent a unique structural state, would bear the potential of formulating structure-property relationships for metallic glasses similar to those that have enabled the success of crystalline alloys. Here we discuss recent advances that nanomechanical testing has contributed with to spatially resolve property fluctuations in metallic glasses from the nanometer to micrometer scale. We highlight the range of correlation lengths reported so far, discuss their possible origin, and demonstrate material-specific and instrument-related challenges along our own and the community's path to experimentally quantify structural length scales of glassy metals.

11:20 "Molecular Dynamics Simulations of Push-To-Pull Tests in Graphene Sheets"

Javier Varillas[1,2,*], Jaroslav Lukeš[3], Otakar Frank2[2]

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Our investigation concerns large-scale molecular dynamics (MD) simulations of graphene employing reactive empirical bond-order interatomic potentials. The graphene sheets are subjected to a set of loading conditions that mimic those in push-to-pull (PTP) micro-scale experiments. In our simulations, we additionally introduce an uneven adhesion distribution between the graphene and the pulling plates whose mutual, weak-bond interaction is described through Lennard-Jones pairwise potentials. Moreover, the analysis includes the role of specific lattice defects (grain boundaries vs. vacancies) that plays in the resulting stress-strain curves as compared to those from bulk-like pristine graphene tensile tests. The results from the MD simulations indicate that the development of atomic-level stress concentrators located at the edge of the plates or along the grain boundaries systematically leads to premature failure of graphene under PTP conditions.

11:40 "Combining Nanoindentation and Simulation for elasto-plastic material data of thin metal films"

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It is known, that the properties of thin metals differ from bulk material due to size effects, and are also highly process dependent in their properties. Still, those properties are urgently required by industry to feed them into lifetime simulations for accurate reliability predictions based on the physics of the involved failure mechanisms. One method to extract elasto-plastic data from nanoindentation measurements is to fit a material law (e.g. Ramberg Osgood) by simulation to reproduce a measured force-displacement curve. This is shown for a 1 µm Al film on Si-substrate.

12:00 "Nanoindentation of thin films – accurate assessment of Young's modulus"

Stanislav Žák[1,*], Megan J. Cordill[1,2]

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The nanoindentation is a modern and progressive tool for assessment of material mechanical properties in a reliable, fast and almost non-destructive way. Recent development in use of thin films applications (such as modern electronics and wearable or bendable devices) leads to high demand for accurate measurement of hardness and elastic modulus of thin metallic films with micron and sub-micron thickness. This can be easily achieved with the nanoindentation, however, a strong emphasis must be placed on proper evaluation of the indentation results to avoid mixing the film and substrate effects. For decades, it was assumed that contact depth of 10% of the film thickness is safe for both hardness and elastic modulus. Our recent research shows that it is true only for the hardness (as a short range property) measurements. For proper evaluation of the thin film elastic modulus (as a long range property), very shallow indents have to be made and combined with more deep ones to separate the substrate effects. Presented work combines experimental nanoindentation with the finite element modelling connecting the indentation process, measured values and evolution of the strain fields in the film substrate system under the indenter tip to show the dependence of the size of influenced area on the indentation depth.

Abstract - Poster Presentations

1. “Mechanical Properties of Interface Adhesion in Solid State Batteries”

Shatha AlMarri[1,*], Ed Darnbrough[1], David Armstrong[1]

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The increased demand for lithium batteries in various applications such as electric vehicles and portable electronics have motivated further battery research to improve energy density, safety, and lifetime. The most promising of these advancements is the solid-state battery, which utilizes a solid electrolyte instead of the conventional organic liquid electrolyte. The replacement of the liquid electrolyte with a solid one however, introduces several complexities at the electrolyte-electrode interface. Weak adhesion at this interface will introduce several voids, which lead to microcracking after several cycles, which ultimately degrades the battery and hinders lithium-ion movement.

In this study two methods, nanoindentation and nanoscratching, are used to characterize the electrode-electrolyte interface and extract the strength of adhesion. 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 for various thin film systems of different thicknesses and materials. Furthermore, delamination energy has been extracted through quasi-static indents by observing the areas under the load-displacement curve. Additionally, the mechanical properties of novel electrolyte polymer candidates have been found through nanoindentation.

2. “Nanoindentation and nanostructures flattening as a tool to obtain surface tension in chalcogenide glass-forming materials”

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The presented contribution focuses on combination of nanoindentation experiments and following of flattening on nanostructures imprinted on a surface of amorphous chalcogenides to evaluate surface tension in undercooled chalcogenide melts. Preparing and following of flattening process of periodical nanostructures on a surface of amorphous materials are important for surface mobility study (near-surface viscosity and surface diffusion). Nevertheless, these studies based on evaluation of rate constant of the flattening process need also knowledge of surface tension of the material. Nevertheless, surface tension of glass-forming materials especially in the region of undercooled melt. In this contribution we will show a combination of nanoindentation measurements providing the near-surface viscosity with measurements of flattening process of periodical nanostructures to evaluate surface tension of the studied materials.

Acknowledgements:

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3. “Nanomechanical study of PLA-based nanocomposite materials”

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Nowadays, the biodegradable polymers as polylactic acid are of great interest for the researchers being a potential alternative to replace the conventional plastic materials that have an essential input in the global environmental pollution. The modification of polymer matrix by incorporation of carbon nanoparticles possessing unique properties is a good strategy to produce novel composite material and at the same time to overcome some drawbacks owned by the neat polymer. Using the low cost but effective melt blending method, a series of nanocomposite specimens with various GNPs and/or MWCNTs loading has been prepared. Improved nanomechanical characteristics of the composite samples, apparently due to the carbon nanofillers acting as reinforcement, were discovered by applying quasi-static nanoindentation, accelerated property mapping, nanoscratch and nanodynamic mechanical analysis. Formation of hybrid nanocomposite structure might be the reason of ascertaining the synergistic effect of GNPs and MWCNTs on particular nanomechanical parameters as nanohardness and reduced elastic modulus.

4. “Advances in combined mechanical and electrical SPM characterization of thin films”

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The presentation discusses recent applications of Scanning Probe Microscopy (SPM) methods and case studies for the combined mechanical and electrical characterization of functional films and semiconductor devices. The contribution includes various methods of SPM-based wear tests in combination with Conductive Atomic Force Microscopy (C-AFM). Case studies for the material and device characterization or defect localization are presented for different thin film materials and systems such as diamond-like carbon (DLC) and various types of dielectrics. Scalpel AFM, a three-dimensional combined mechanical and electrical characterization method is discussed. In principle it is a contact mode AFM operation with elevated contact force. By adapting the deflection setpoint, the tip slightly penetrates the sample material and removes it within the given scan area. A sequential repetition of a removal scan with elevated setpoint and a read scan with low deflection setpoint across a larger scan area enables three-dimensional material characterization with known z-steps. By simultaneously applying a sample bias in C-AFM mode, this technique ena-bles the three-dimensional observation of e.g., conductive filaments in oxide based resistive random-access memory (ox-RRAM) cells. They consist of metallic top and bottom electrodes with a dielectric in between. The application of an electric field leads to the reversible formation of conductive filaments that bridge bottom and top electrode and enable current to flow. Because devices’ performance is strongly related to the dynamical shape, position, and amount of such filaments, Scalpel AFM is a promising tech-nique to study the conduction mechanism in RRAM cells with nanoscale resolution.

5. “Correlative Characterisation of Rangifer Tarandus (Reindeer) Antler, Evaluating Differences Between Male and Female and Calving Females”

Rachel Board[1], Elizabeth Sackett[1], Ude Hangen[2], Jebin Jestine[1], Richard E Johnston[1]

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Antlers are large, biomineralized bony structures that emanate from deer skulls. These extraordinary features usually grow on male deer species and are used in the rut as they compete for dominance to mate. The exception to this is the species *Rangifer tarandus* (Reindeer) where the females also possess antlers. Male reindeer shed their antlers after the rutting season in autumn whereas the female reindeer keep their antlers throughout the winter and will use their antlers to defend patches of food against other reindeer. In the spring, after the calving season, the antlers are shed. Previous comparative studies of male and female antlers have recommended further analysis into mechanical properties. In these previous studies, the maternity status of the females was not considered. This study aims to comprehensively characterise reindeer antlers by correlating multiple techniques from X-ray micro-CT to assess the internal microstructure, XRF and SEM-EDS for analysis of the mineral components as well as nanoindentation to evaluate the nanomechanical properties of each of the three groups (male, female, and female with calf). Between properties of the male and female, without calving antlers, there is very little difference in the modulus and hardness values. However, a difference in average hardness between the females with calves to the male and females without calves was lower, the females with calves also had an average lower modulus than their counterparts. Chemical analysis also showed that females with calves had an average lower phosphate concentration compared to the male and female reindeer.

6. “Local determination of stress-strain curves for multi-material semi-finished products made by laser build-up welding”

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Multi-material solutions offer numerous benefits as they, in contrary to conventional monolithic parts, provide for the adjustment of locally adapted properties. One manufacturing approach for hybrid components, which is being investigated in the Collaborative Research Centre (CRC-1153) “Tailored Forming”, is the use of pre-joined hybrid semi-finished products. One approach to the production of hybrid semi-finished products investigated in this context is build-up welding. A central challenge in the use of such semi-finished products is the determination of material properties of each material after welding. The resulting joining zones are only a few micrometres thick and cannot be characterised using conventional methods of investigation. Therefore, a method is developed to determine strain-stress curves from experimental Nanoindentation tests with a spherical tip. The investigated hybrid semi-finished product consists of a base material and two laser build-up welded layers of 1.4430 and 1.4817. The semi-finished product was produced by applying a few layers of 1.4430 on the base material by laser build-up welding. Subsequent, additional layers of 1.4818 was applied also by means of laser build-up welding. Using the developed methodology stress-strain curves were extracted for every laser build-up layer of each material layer and compared to each other. Particular interest was given to the area of material transitions. In further investigations, the material properties for forged components consisting of the same layer system are also to be determined using the developed methodology. Comparison with the measured stress-strain curves of the hybrid semi-finished products will allow the investigation of a change in properties as a result of forming.

7. “Comparison of methods for nanomechanical characterization of soft specimens”

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Different soft hydrogels have found applications in biological science due to their biocompatibility, flexibility and non-toxicity. For the nanoscale material property analyses atomic force microscopy (AFM) methods has been widely used even the specimens are placed in various environments. The novel AFM imaging modes, based on the periodic contact of the cantilever tip to the sample surface, allow sufficiently precise force control, thus it is possible to minimize the force at which AFM tip interacts with the specimen. In such modes the peak interaction forces and nanoscale properties are collected for each individual tap simultaneously. Peak Force Quantitative Nano Mechanics mode was applied to determine soft hydrogels, prepared on Si substrates elastic properties. The attractive features of AFM are associated with the complexity of the experimental set-up and demanding analyzes of collected specimen data. Hydrogels and also organic polymers are much more compliant compared to the typical semiconductor materials, with the elastic modulus values down to MPa and kPa range. Nowadays, similarly to AFM, the nanoindentor for multiscale quantitative mechanical testing of soft materials and bio samples operating in buffer fluids has been developed. Our goal was to firstly use the unique combination of force sensitivity and displacement range of the in-situ indenter BioSoft to perform indentation measurements on organic polymer materials which have been typically used in organic field effect transistors (OFET’s). This work was financially supported by the Slovak Research and Development Agency under the contract No. APVV-18-0480.

8. “Investigation of the adhesion of molybdenum and molybdenum trioxide to cube corner indenters by nanowear tests”

Norman Heimes[1,*], Bernd-Arno Behrens[1], Gerhard Poll[2], Dennis Konopka[2], Hendrik Wester[1], Florian Pape[2]

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Due to the ecological change, environmentally friendly and innovative solutions are more and more in demand. Solid lubricants can replace oils and greases as lubricants in rolling contacts. The current research project is concerned with the investigation of a regenerative dry lubricant based on molybdenum. In this process, molybdenum trioxide (MoO_3) is generated continuously by tribo-oxidation from a molybdenum (Mo) coating. To ensure that MoO_3 is already available during the running-in phase, a thin MoO_3 top layer is applied to the Mo coating. The lubrication potential of MoO_3 was demonstrated in bearing rig tests. In addition, it was found that transfer lubrication occurred, from the bearing washer to the uncoated rolling element. To analyse the wear of the individual layers, nano wear tests were carried out and a wear model was transferred [1]. In order to further align the boundary conditions of the nano wear test with the bearing tests, it was investigated whether a transfer from the coatings to the cube corner indenter occurred. For this purpose, two cube corner indenters were examined after the wear test for Mo and MoO_3 using SEM. It was found that particles adhered to both indenters, which were subsequently confirmed as Mo and O by EDX analysis. In addition, the adhesions on the indenter tested for MoO_3 were significantly larger than on the indenter tested for Mo. It could be shown that a transfer formation of Mo and MoO_3 from the indenter also took place in the nano wear test.

[1] Behrens B.-A., Poll G., Möhwald K., Schöler S., Pape F., Konopka D., Brunotte K., Wester H., Richter S., Heimes N., Characterization and Modeling of Nano Wear for Molybdenum-Based Lubrication Layer Systems. *Nanomaterials*, **11**, 1363, 2021.

9. “Effect of combination of carbon nanofillers on the nanomechanical properties of PLA-based composites films”

Evgeni Ivanov[1,2,*], Todor Batakliev[1,2], Giovanna Buonocore[3]

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Polymers and its composites have generally lower wear and scratch resistance than other materials and therefore these properties must be carefully controlled. Nanoindentation and scratch testing is one of the methods that allow well defined characterization of resistance of polymeric materials, both bulk and coatings. The polymer used in this study for preparation of nanocomposites was Ingeo™ Biopolymer PLA-3D850. Two types of nanoparticles were chosen as nanofillers: 1) Graphene TNGNP; 2) Multiwalled carbon nanotubes (MWCNTs). The mono (PLA/MWCNT and PLA/GNP) and bifiller nanocomposites (PLA/MWCNT/GNP) made by combining both fillers in different proportions were processed using the melt extrusion method trough preparation of masterbatches and further dilutions. XPM (fast indentation or accelerated property map) and nanoscratch measurements were made on Hysitron TI 980 instrument (Bruker, MN, USA) by using 2D transducer assembly (both normal and lateral force) equipped with Berkovich probe. The results from nanoindentation shows that the sample filled with 6 wt.% of MWCNTs have a maximum improvement with 25% and 43% for the hardness and Young’s modulus. The samples with hybrid combination of both nanofillers MWCNTs and GNP (1:3, 1:1 and 3:1), also shows improvement. Synergy effect with combining of GNPs and MWCNTs is found pointing out higher scratch resistance of the bifiller composites, compared to monofiller composites with same carbon loading.

Acknowledgments:

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10. “Different approaches for determination of the mechanical behavior changes in micro-structured glasses”

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Heavy Metal Oxide (HMO) glasses represent a special kind of oxide glasses containing a large amount of oxides of heavy metals such as PbO, Ga_2O_3, Bi_2O_3. In our previous studies [1, 2] we used direct laser writing technique for the micro-structuring of their surface by the formation of the microlenses, planar optical waveguides or microcraters. The different modes were used for determination of the changes in their mechanical behavior: force spectroscopy and nanoindentation set-ups on the AFM and employing classical nanoindentation apparatus. In this contribution, we compare the results of different approaches significantly differing with applied forces and we deduce the consequences of the mechanical changes to the optical parameters of glasses as refractive index.

[1] Smolík, P. Knotek, J. Schwarz, E. Černošková, P. Janíček, K. Melánová, L. Zárybnická, M. Pouzar, P. Kutálek, J. Staněk, J. Edlman, L. Tichý, 3D micro-structuring by CW direct laser writing on PbO-Bi_2O_3-Ga_2O_3 glass, *Applied Surface Science*, **589** (2022) 152993.

[2] J. Smolík, P. Knotek, J. Schwarz, E. Černošková, P. Kutálek, V. Králová, L. Tichý, Laser direct writing into PbO-Ga_2O_3 glassy system: Parameters influencing microlenses formation, *Applied Surface Science*, **540** (2021) 148368.

11. "Local mechanical properties testing of zirconium alloy nuclear fuel claddings"

Ondřej Libera[1,*], Jan Procházka[1], Patricie Halodová[1], Petra Gávelová[1], Jakub Krejčí[2]

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Zirconium alloys are being commonly used as a material of choice for nuclear fuel claddings in water cooled nuclear reactors for decades due to their good corrosion resistance and low neutron absorption. However, the increasing operation conditions of next generation nuclear reactors (Gen-IV) in terms of higher temperatures, pressures and higher neutron flux requires evaluation of further Zr cladding usability. The embrittlement of Zr claddings due to hydrogen pickup from reactor coolant is one of the issues for its potential use in Gen-IV reactors. Nanoindentation is an effective tool for analysis of the change of mechanical properties of hydrogen enriched Zr claddings from localised material volume. Zirconium alloy Zr-1Nb (E110) with experimentally induced hydrides was analysed by the means of nanoindentation. Zirconium hydrides were formed in the material after exposure in high temperature water autoclave. The nanoindentation measurements were performed as an array of 10x10 indents across areas with hydrides. Depth dependent mechanical properties measured by nanoindentation were compared between the material with no hydrogen content, low hydrogen content (127 ppm H) and high hydrogen content (397 ppm H). Another set of nanoindentation measurements were performed on Zircalloy-4 (Zr-1.5Sn-0.2Fe-0.1Cr) cladding tubes after high-temperature oxidation (950°C – 1425°C) in steam, simulating severe accident conditions in pressurised water reactors (PWRs). Linear nanoindentation profile measurements were conducted in radial direction of the cladding across ZrO_2 layer, oxygen enriched α-Zr phase, α-β Zr transition phase and into base β-Zr material. The change of mechanical properties relates to oxygen concentration profiles obtained by wavelength dispersive spectroscopy (WDS) measurements.

12. "Atomic Force Microscopy of Biosamples"

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The Atomic Force Microscope is not just an imaging device. It also provides information about the mechanical properties of samples through highly localized nanoindentation. Our laboratory is performing research in the field of imaging of biomolecules [1], mapping the elastic properties of cells and their clusters [2], and characterization contractile properties of cardiomyocytes and their clusters [3]. The poster will present these main applications, but also the equipment of the laboratory, which offers services to scientific groups as well as to other interested parties.

The laboratory's flagship is the large AFM microscope JPK NanoWizard 4XP installed on a Leica DMi8 optical microscope with a fluorescence module. Imaging combined with mapping of elastic properties can be performed under semi-physiological conditions. Combining the AFM with microfluidic, so-called FluidFM enables the possibility to aspirate and/or deliver extremely low volumes. This feature can be used when injecting or removing small volumes from individual cells.

Keeping on the cutting-edge current AFM technology, the new generation of the MultiMode AFM microscope, version 8HR, was built for imaging with the maximum resolution that current commercial setups allow. This AFM setup will help the structural biologist image the biomolecules (DNA, proteins, molecular complexes) on a single molecular level.

Acknowledgements:

We acknowledge CF Nanobiotechnology of CIISB, Instruct-CZ Centre, supported by MEYS CR (LM2018127).

[1] Horňáková, V., Přibyl, J. & Skládal, P: *Monatsh Chem* **147** (2016) 865–871.

[2] Raudenska M., et al.: *Scientific Reports* **9** (2019) 1660, 1-11.

[3] Caluori G, et al.: *Biosens Bioelectron.* 124-125 (2019) 129-135.

13. "Surface properties of MXene sprayed films"

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MXenes, as a new family of 2D materials, were discovered in 2011 [1]. In this work we focused to characterization of surface and mechanical properties of MXene films, prepared by spray coating method onto glass substrate. With this method, we are able to coat relatively big areas of different substrates and prepare homogenous, thin and smooth films. Mechanical properties of prepared films were analysed by Nanoindentor TI 750 Ubi (Hysitron, USA). This technique also provides the ability to analyze the surface topography using SPM mode (Scanning probe microscopy).

SEM images confirmed that using a MILD method for MXene etching, single-layer sheets of MXenes were prepared with a diameter of about three μm. XPS confirmed structure of prepared MXene. Using For 5 layers MXene sample average thickness of 36.80 nm and for 10 layers MXene samples 50.62 nm was calculated by SPM. For indentation the partial load-unload curves with 50 segments and maximal load 1 mN was used. Elastic moduli of 5 layers and also 10 layers films were very similar, 89.81 GPa and 89.43 GPa, and higher as of pure glass, 86.73 GPa. The hardnesses showed greater differences 10.50 GPa for 5 layers films, 7.46 GPa for 10 layers films and 9.19 GPa for pure glass samples.

Knowledge about the macromechanical properties of these new material MXenes is important for many applications.

Acknowledgments:

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14. "Modified consolidants of porous materials with multifunctional performance"

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Even if silicon alkoxides (especially ethylsilicates) have long been used as consolidants of weathered stone monuments, their physical properties are not ideal. In this study, an innovative procedure for the consolidation of sedimentary rocks was developed. Combining the use of organometallic and alkylamine catalysts and the addition of well-defined nanoparticles ethylsilicate gels were prepared which exhibit mesoporosity and whose cracking was suppressed. Using the developed consolidation procedure, the mechanical and surface properties of the rocks were improved without the unwanted over-consolidation of the surface layers of the stone, and any significant deterioration in the pore size distribution. In order to minimize the potential negative impact of used nanoparticles, their toxicity was determined. Based on the outcome of the toxicological study, those with the optimum performance with minimum toxicity were selected.

15. "Development of a push to pull microsystem for tensile strength tests with cells of filamentous microorganism"

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Filamentous microorganisms are widespread in industrial biotechnology as production systems, for example of active pharmaceutical ingredients or organic acids. Depending on the cultivation conditions, biomass growth takes place as dispersed mycelium, as clumps or in the form of dense bioagglomerates (pellets). Especially shear stress, induced by the fluid in stirred bioreactors, determine the morphology. Depending on the strength of the mechanical stress, individual tube-like cells (hyphae) can be sheared off in the outer region of the pellet (erosion) or an entire pellet can break. In this context, the tensile strength of the hypha is an important property that is currently difficult to determine experimentally. For this reason, a push-to-pull microsystem was designed and manufactured to perform tensile tests with an individual hypha. For this, a hypha is glued to the microsystem in liquid above a gap using a microscope with two micromanipulators. Afterwards, the microsystem is placed vertically in a water filled reservoir in the nanoindenter (Hysitron/ Bruker). By applying an increasing load to the microsystem with a flat punch the internal springs are deflected, which leads to an opening of the gap and thus a stretching of the hypha. The mechanical response of the hypha is determined from the comparison of two force displacement curves before and after a fracture event. In the future, this method can be utilised for the comparison of different biological strains or for the generation of input parameters needed for modeling and simulation of pellet fragmentation.

Acknowledgments:

This work has been supported by the Ministry of Health of the Czech Republic [grant number NU21-06-00356.

[1] J. Jezek, J. Sepitka et. al, The role of vascularization on changes in ligamentum flavum mechanical properties and development of hypertrophy in patients with lumbar spinal stenosis. *Spine J.* **20** (2020) 1125-1133.

16. "Micromechanical Properties of Native Human Ligamentum Flavum"

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Ligamentum flavum (LF) is a ligament located in the spine canal and the changes in this ligament significantly contribute to the development of lumbar spinal stenosis (LSS), which is a serious and relatively common disease in the aging population. LSS often leads to disability. There is no prophylaxis of this disease in current knowledge yet. Only one study shows significantly inferior stiffness of LF in cases with LSS. Although it is natural to consider that the mechanical properties of ligamentum flavum can be altered due to ligamentous hypertrophy, only few studies have mentioned on this point [1]. However, to date, there are no reports describing local micromechanical properties of ligamentum flavum depending on anatomical localization. The tissues used for analysis of micromechanical properties were sectioned into 1-2 mm thick sections using microtome blade and glued using Epiglu on the Petri dishes. All testing was performed submerged in 1x PBS containing 1% (v/v) Protease Inhibitors. Quasistatic microindentation of samples was performed to obtain loading curves from the cuts on the cranial, medial, and caudal part of each ligamentum flavum. Average elastic moduli were in the range from units kPa up to hundreds of kPa. We described the local micromechanical properties of ligamentum flavum depending on anatomical localization. We found the gradients of the mechanical properties across the cranial, medial, and caudal cuts of native ligamentum flavum. Medial part of ligamentum is significantly stiffer than cranial and caudal. Central region of cranial, medial, and caudal cut is significantly stiffer than outer region.

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This work has been supported by the Ministry of Health of the Czech Republic [grant number NU21-06-00356.

[1] J. Jezek, J. Sepitka et. al, The role of vascularization on changes in ligamentum flavum mechanical properties and development of hypertrophy in patients with lumbar spinal stenosis. *Spine J.* **20** (2020) 1125-1133.

17. "Development of protocols for measuring twinning stress using micropillar compression"

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The equiatomic CoCrFeNiMn high entropy alloy (HEA), also known as Cantor alloy, has been widely studied due to its outstanding combination of mechanical properties, especially under cryogenic temperatures. The mechanical behavior observed under such conditions, i.e. increase in ductility and tensile strength, was attributed to deformation by extensive mechanical twinning due to their low stacking fault energy. Activation of twinning was also observed in room temperature conditions, although requiring high strain. Given the importance of twinning as a deformation mechanism for the Cantor alloy, further investigations are necessary to better understand the mechanism individually. Therefore, in this work we developed protocols to assess critical resolved shear stress (CRSS) for deformation twinning of Cantor alloy by using in situ micropillar compression. Specific single slip grain orientations and different micropillar sizes were chosen aiming to observe transition from full to partial dislocations. Three different micropillars sizes (1, 0.5 and 0.3μm diameter) were micromachined by focused ion beam (FIB) and mechanically analyzed by uniaxial *in situ* micropillar compression. Subsequently, post-mortem imaging was performed to identify the slip systems activated during testing. The results provided quantitative insights essential for further understanding of the twinning mechanisms, also valuable inputs for alloy design to enhance deformation twinning in HEAs.

18. "Near-surface viscosity in amorphous chalcogenides"

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This contribution focuses on the study of the near-surface viscosity of glass-forming chalcogenides in the form of bulk samples and thin films, using various experimental methods to provide reliable data for comparing the viscosity in bulk and near-surface of amorphous materials. One of the possible methods to study near-surface viscosity is measurement of flattening of micro- and nanostructures imprinted on the surface of samples. [1] The flattening process is caused by movement of the structural units and can be followed by AFM. Change in the structure imprinted on the surface of a sample. Another suitable method to study near-surface viscosity is nanoindentation [2, 3]. During nanoindentation, a defined force is applied to a small tip of a defined shape and pressed into the surface of the sample. After penetration of the indenter, the force load can be immediately released or kept constant for a certain time. Nanoindentation is a very suitable method for measuring viscosity. From the dependence of the square of the penetration depth on time, the viscosity value can be determined. [4] The advantage is that no other quantity, such as surface tension, needs to be known for the data evaluation. Therefore, it is a universal method suitable for any material. This contribution will be focused on viscosity measurements in thin films and in surface of bulk samples using nanoindentation for $Ge_{25}Se_{75}$ and $As_{20}Se_{80}$ compositions. The viscosity values obtained are then compared with those obtained by other methods in bulk samples.

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Practical Information

Nanobrücken 2022

Nanobrücken 2022 will be held at the Charles University, located in the beautiful city of Prague.

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 https://www.bruker.com/Nanobruecken.

Oral Presentation Guidelines

The workshop prefers that you use your own laptop computer. However, bringing a backup of your presentation file on a memory stick is recommended. 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

Charles University, Karolinum
Ovocny trh 5, 11636 Praha 1, Czech Republic (Entrance via Celetná 20)