Nanobrücken 2024

A Nanomechanical Testing Conference
and Bruker User Meeting

March 19 - 21, 2024 | Valpré-Lyon | Lyon, France

Nanobrücken 2024 Program

Preface

Bruker is pleased to announce that **Nanobrücken 2024** will take place March 19-21 in the beautiful city of Lyon.

This is the 14th 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

Hôtel Valpré-Lyon
1 Chemin de Chalin
69130 Ecully, France

Program Committee

Conference Committee Members

Dr. Lucile Joly-Pottuz, INSA Lyon (France)
Prof. Karine Masenelli-Varlot, INSA Lyon (France)
Prof. Denis Mazuyer, Ecole Centrale de Lyon (France)
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Dr. Rhys Jones, Bruker (United Kingdom)
Jérôme Beaumale, Bruker (France)
Annette Niedballa, Bruker (Germany)

Day 1: Tuesday, March 19

Welcome

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

Talks — Session I

Chair: Douglas Stauffer, Bruker

13:45 "Nanoindentation at the Elastic Response Limit: Revealing Elastic Microstructures in a Monolithic Glass"
Reza Rashidi, Federal Institute of Materials Research and Testing (BAM)

14:00 "Local mechanical properties of $BiFeO_3$ ceramics"
Katarina Ziberna, Jožef Stefan Institute

14:15 "Quantitative mechanical characterization at the nanometer scale using AFM PeakForce™ QNM™ mode in the spherulitic structure of a semi-crystalline polymer"
Jérémy Grondin, Université de Poitiers, Institut Pprime

14:30 "Effect of electron irradiation on silicate glasses plasticity during in-situ micromechanical tests"
Matthieu Bourguinon, PSL University, CNRS & Sorbonne University

14:45 "Novel approach to wear modelling in the nano-scale for molybdenum-based lubricant coating systems"
Norman Mohnfeld, Leibniz Universität Hannover

15:00 "FEM study of the validity domain of two analytical nanoindentation models for thin film elastic modulus extraction"
Marina Melo de Lima, STMicroelectronics & Univ. Grenoble Alpes

15:15 Coffee Break

Talks — Session II

Chair: Melanie Gauvin, OCAS Microscopy Centre

15:45 "Structure-Property Relationships of 3D/4D Printed Microstructures"
Clara Vasquez-Martel, Heidelberg University

16:00 "Investigation of the Deformation Behavior in a CoCrNi Medium Entropy Alloy with Short-Range Order Structure via In-Situ Compression Transmission Electron Microscopy"
Tai Cheng-Ling, National Taiwan University

16:15 "Micromechanical characterization of NMC cathodes from Li-ion batteries"
Tobias Sedlatschek, RWTH Aachen University

16:30 "In-situ XRM study of fracture behaviour of on-chip interconnect stacks with complementary FEM analysis"
Stefan Weitz, Fraunhofer IKTS & Brandenburgische Technische Universität

16:45 Short Break

Talks — Session III

Chair: Lucile Joly-Pottuz, INSA Lyon, Universite Claude Bernard Lyon 1, CNRS

17:00 "New geometry for shear properties measurement and high strain rates testing at micro-scale"
Benedicte Adogou, Ecole de Mines de Saint-Etienne

17:15 "A nanoindentation study on the mechanical behavior of the cementum-dentine junction in wild boar mandibular molars"
Ahmed Ali, Technische Universität Berlin

17:30 "Combining XRD and nanoindentation to characterize mechanical properties evolution of flax cell walls during controlled heat treatment"
Celia Caër, ENSTA Bretagne, UMR CNRS 6027, IRDL

17:50 "TI990 - the next generation"
David Vodnick & Rhys Jones, Bruker

Poster Session

All Posters are Eligible for Top Poster Prize

18:10 Poster Session & Welcome Reception (all posters, see list on page 7)

Keynote Lecture

Chair: Brad Boyce, Center for Integrated Nanotechnologies, Materials Science and Engineering Center, Sandia National Laboratories

19:45 "**Combining nanomechanical testing with AFM and TEM to study small scale plasticity: application to MAX phases**"
Prof. Christophe Tromas, Université de Poitiers, Institut Pprime

Day 2: Wednesday, March 20

Talks — Session IV

Chair: Oden Warren, Bruker

09:00 Opening
Lucile Joly-Pottuz & Karine Masenelli-Varlot, INSA Lyon, Denis Mazuyer, Ecole Centrale de Lyon & Oden Warren, Bruker

Invited Talk

09:15 "Spherical Nanoindentation Testing – a promising way to measure localized flow curves"
Verena Mayer-Kiener, Montanuniversität Leoben

09:45 "Inverse method to determine cyclic properties of materials by combining cyclic indentation and numerical simulation"
Hafiz Muhammad Saijad, Ruhr-Universität Bochum

10:05 "Time-dependent mechanical behavior of semicrystalline polymers by multi-scale characterization.
Julie Pepin, Univ. Tours, Univ. Orleans, INSA CVL

Invited Talk

10:25 "New Insights into the Statistical Nanoindentation Methodology"
Esteban Broitman, SKF Research and Technology Development Center

10:55 "Strain Rate Dependent Crystal Plasticity Parameter Optimization for CPFEM Nanoindentation Simulations in Single Crystals"
Rongfei Juan, Aalto University

11:10 Coffee Break

Talks — Session V

Chair: Joan Josep Roa, Steros GPA Innovative S.L

11:40 "New leads on the densification profile underneath nano indentation imprint in silica glass by means of a chemical dissolution technique"
Jean-Pierre Guin, Univ. Rennes 1, UMR CNRS 6251, IPR

12:00 "Exploring chemomechanical weakening in muscovite via liquid nanoindentation"
Frank DelRio, Sandia National Laboratories

12:20 "Irreversible evolution of dislocation pile-ups during cyclic microcantilever bending"
Szilvia Kalacska, Mines Saint-Etienne, Univ Lyon, CNRS

Invited Talk

12:40 "Quantification of electron-irradiation creep in amorphous olivine and amorphous silica from In situ TEM and SEM nanomechanical testing"
Guillaume Kermouche, Mines Saint-Etienne, Laboratoire Georges Friedel

Lunch Break

13:10 Lunch Provided On-Site

Talks — Session VI

Chair: Karine Masenelli-Varlot, INSA Lyon

Invited Talk

14:30 "In-situ TEM straining experiments to probe dislocation mechanisms"
Frederic Mompiou, Chercheur CNRS, CEMES

15:00 "A Novel combination of lab-on-chip testing, nanoscale DIC, and ACOM-TEM for unraveling plasticity mechanisms in UFG freestanding metal thin films"
Ankush Kashiwar, IMAP-IMMC, Université catholique de Louvain

15:20 "Automated co-localized imaging and analysis for high-throughput in-situ nanoindentation – Hysitron PI 89 Auto SEM PicoIndenter"
Sanjit Bhowmick, Bruker

15:40 "Nanomechanical testing of argyrodite particles for solid battery electrolyte applications"
Johannes Ast, CEA Liten

16:00 "Understanding the micromechanical deformation of anodes, cathodes and solid electrolytes in solid state lithium ion batteries"
David Armstrong, University of Oxford

16:20 Coffee Break

Talks — Session VII

Chair: David Armstrong, University of Oxford

16:50 "Enriching nanoindentation with in-situ electrical measurements and SEM observations"
Fabien Volpi, SIMaP, Université Grenoble Alpes

17:10 "Eliciting stable nanoscale fracture in single-crystal silicon"
Douglas Stauffer, Bruker

17:30 "In-situ scanning probe microscopic (SPM) Imaging of Cu pumping phenomenon in hybrid bond wafers"
Ali Roshanghias, Silicon Austria Labs GmbH

Invited Talk

17:50 "Statistical indentation - and correlated microscopy techniques"
Brad Boyce, Center for Integrated Nanotechnologies, Materials Science and Engineering Center, Sandia National Laboratories

18:20 End of Day

19:30 Conference Dinner

Day 3: Thursday, March 21

Talks — Session VIII

Chair: Denis Mazuyer, Ecole Centrale de Lyon

Invited Talk

09:00 "Nanomechanical testing of steels using nanoindentation"
Melanie Gauvin, OCAS Microscopy Centre

09:30 "Local mechanical properties of oxide inclusions"
Sandor Lipscei, École Polytechnique Fédérale de Lausanne

09:50 "Correlation at the sub-micrometric length scale between surface integrity and microstructure in WC-Co grades"
Joan Josep Roa, Steros GPA Innovative S.L

10:10 "Friction and roughness at the nanoscale"
Juliette Cayer-Barrioz, LTDS, CNRS UMR5513 Ecole Centrale de Lyon

10:30 "Identification of the mechanical and electrical behaviors of a copper oxide layer by resistive-nanoindentation tests"
Muriel Braccini, Univ. Grenoble Alpes, CNRS

10:50 Coffee Break

Talks — Session IX

Chair: Guillaume Kermouche, Mines Saint-Etienne, Laboratoire Georges Friedel

Invited Talk

11:20 "Tribology and materials memory effects in polymers.(ten.)"
Antoine Chateauminouis, SIMM, ESPCI / CNRS

11:50 "Fretting behavior of an electrical contact"
Dominik Linsler, Fraunhofer Institute for Mechanics of Materials IWM

12:10 "Modeling the squeezed-thin film accounting for adsorbed nanometric monolayers"
Denis Mazuyer, École Centrale de Lyon, LTDS UMR 5513 CNRS

12:30 "Tribochemistry of Sulfur Containing Additives on DLC coatings: Contribution of Electronic Spectroscopies"
Aslihan Sayilan, Ecole Centrale de Lyon& INSA de Lyon

12:50 "The interest of nanoscale tests in the prediction of tribological behavior of dynamic system"
Houcine Ben Abdelounis, Tribology & Materials for Industry

13:10 Farewell – Organizing Committee

13:20 Farewell snack: Saying goodbye with a delicious treat

Poster List

1. "Multi scale in-situ micromechanical testing of Polymer Core Solder Ball (PCSB) coatings for BGA interconnections"
   Sergio Sao-Joao, Mines Saint-Etienne, Univ Lyon, CNRS
2. "Obtaining of Bulk Ultrafine Grained Multicomponent FeWAlTiNi Composite Materials by Ball Milling and Explosion Densification Technology"
   Nikoloz Chikhradze, LEPL Grigol Tsulukidze Mining Institute & Georgian Technical University
3. "An equivalent homogenized finite element model of TiAl/TiAlN multilayered coatings"
   Sylvain Giljean, Université de Haute-Alsace
4. "Determining The Flexoelectric/Photo-Flexoelectric Response of Strontium Titanate Cantilevers by Nanoindentation Instrumentation"
   Michal Budzialowski, Adam Mickiewicz University
5. "Characterization of surface and mechanical properties of microbial cells"
   Marketa Khyrova, Brno University of Technology & Czech Academy of Sciences
6. "A robust and fast method to estimate cubic elastic constants based on nanoindentation and Bayesian Inference"
   Jean-Sebastien Lecompte, Université de Lorraine
7. "Deformation mechanism of cerium oxide nanocubes studied by in situ nanocompression in environmental TEM"
   Lucile Joly-Pottuz, INSA Lyon, Universite Claude Bernard Lyon 1
8. "Quantification of Irradiation Damage in Nuclear Power Plant Structures through Indentation Size Effect Analysis"
   Qamar Hayat, Coventry University & The University of Warwick
9. "Formation of nanoscale intergranular cavities: in situ SEM and simulations"
   Dome Tanguy, Université Claude Bernard Lyon 1
10. "Creating a digital twin and how it helps to speed up your coating development"
    Nick Bierwisch, Saxonian Institute of Surface Mechanics (SIO)
11. "Towards bulk and surface mechanical properties of a polyimide sample using AFM and instrumented nano-indentation"
    Thomas Jalabert, Univ. Grenoble Alpes, CEA
12. "Investigation of the elastic properties of single Au nanoparticles by the coupling of insitu TEM nanocompression experiments and Brillouin spectroscopy"
    Mariana Timm, INSA Lyon, Université Claude Bernard Lyon 1 & Université de Lyon
13. "Deformation mechanisms of inorganic fullerenes used as lubrication additives : an in situ TEM nanocompression study"
    Pattamadai Sundaram Sankara, INSA Lyon, Universite Claude Bernard Lyon 1

14. “Static friction at the microscale investigated at the limit of Hertzian pressure by oscillatory shear experiments”
    Ahmed Uluca, Trinity College Dublin

15. “Nanoindentation of a rough surface”
    Yvan Maturenet, LAMIH UMR CNRS 8201, Université Polytechnique Hauts de France

16. “Key parameters affecting the local material characterization of interfaces in polymer blends: dialogue between AFM measurements and FEA simulations”
    Mateo Saint Ourens, Université Claude Bernard Lyon 1, INSA Lyon

17. “How to perform micro-bending beam push and pull fatigue during electrochemical hydrogen charging in scratch test mode”
    Lukas Hasenfratz, Saarland University

18. “Measurement of resistance in solid laden liquids – Initial traverses at room temperature”
    Karlis Agris Gross, Riga Technical University

Abstracts - Oral Presentations

Day 1: Tuesday, March 19th

Talks — Session I

13:45 "Nanoindentation at the Elastic Response Limit: Revealing Elastic Microstructures in a Monolithic Glass"

Reza Rashidi[1*], Birte Riechers[1], Robert Maass[1, 2]

1 Federal Institute of Materials Research and Testing (BAM), Unter den Eichen 87, 12205 Berlin, Germany;
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Metallic glasses (MGs) have been shown to be structurally heterogeneous at the nanometer (nm) scale by nanomechanical methods such as atomic force microscopy and nanoindentation. In addition, elastic property mapping has indicated the presence of at least an order-of-magnitude larger length scales, challenging existing theories about the origin of such elastic fluctuations. Which mechanisms could explain the formation of such a hierarchical landscape of elastic heterogeneities? After successfully excluding chemical fluctuations as a possible source of the emerging elastic fluctuations in our previous work (Materials & Design 229 (2023) 111929), we proposed that cooling constraints during solidification may be underlying the elastic long-range fluctuations. To test this hypothesis, we here present a series of nanoindentation experiments, identifying how sample geometry and thermal processing during casting affect elastic correlation lengths in MGs. Our research indicates that these factors are strongly connected to the development of tailored elastic microstructures, which in turn may have significant implications for the material's macroscopic properties.

14:00 "Local mechanical properties of $BiFeO_3$ ceramics"

Katarina Žiberna[1,2,*], Maja Koblar[1], Micka Bah[3], Franck Levassort[3], Goran Dražić [1,2,4], Hana Uršič[1,2] and Andreja Benčan[1,2]

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Ferroelectric perovskites are notorious for their brittleness, which is partially caused by the depletion of dislocations. In recent years, however, dislocations have begun to be intentionally introduced into perovskites by mechanical deformation to tailor their macroscopic piezoelectric, ferroelectric and mechanical properties. This has revealed a gap in the knowledge of the mechanical properties and deformation of these materials at macroscopic and local scales. In this work, we report on the nanomechanical characteristics of $BiFeO_3$ ferroelectric ceramics explored by in situ nanoindentation at room-temperature in the force range from 200 μN to 2 mN. The hardness and the reduced Young's modulus decrease from (9.9 ± 0.4) GPa and (113.7 ± 6.9) GPa, respectively, with increasing force, which we attribute to the structural and microstructural features of the material. In addition, atomic force microscopy was used to detect changes in the Young's modulus of domain walls - nanoscale structural features characteristic of ferroelectrics. The choice of the force range in which the material transitions from ductile to brittle behavior also enabled tracking the progression of plastic deformation under the cube corner indentation probe. A combination of first pop-in analysis in the indentation curves, scanning electron microscopy and scanning transition electron microscopy revealed the sequence

of localized deformation under the probe, starting with homogeneous dislocation nucleation by activation of the room-temperature perovskite slip system $\{110\}_{pc}\langle 1\bar{1}0\rangle_{pc}$ (pc-pseudocubic), dislocation multiplication and movement, and grain subdivision in the proximity of the indented surface, which acts as a center of built-up stress for crack formation.

14:15 "Quantitative mechanical characterization at the nanometer scale using AFM PeakForce™ QNM™ mode in the spherulitic structure of a semi-crystalline polymer"

Jérémy Grondin[1,*], Olga Smerdova[1], Sylvie Castagnet[1] and Christophe Tromas[1]

1 Institut Pprime (UPR 3346 CNRS / ISAE-ENSMA / Université de Poitiers), Department of Physics and Mechanics of Materials, Poitiers, France

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Semi-crystalline polymers are heterogeneous materials characterized by different structural scales. At the micrometer scale, they are characterized by a quasi-spherical spherulitic microstructure, with spherulites organized in radial branches composed of nanoscale crystalline lamellae stacks separated by amorphous phase. Understanding the relationship between the nanoscale lamellar structure and its mechanical properties is essential to better understanding the material's behavior at higher scales. However, conventional instrumented (nano)indentation techniques lack the spatial resolution required for mechanical testing at this scale. Recent developments in nanomechanical testing using Atomic Force Microscopy (AFM) offer imaging capabilities alongside small-scale indentation tests. However, this approach presents challenges due to the complexity of simultaneously accessing the fine nanostructure and the accuracy of mechanical measurements. This is due to the technique's sensitivity to surface topography — which cannot be avoided in the case of semi-crystalline polymers, as the crystalline lamellae organization induces a nanometer-scale surface topography. Overcoming this limitation often involves using larger AFM probes for mechanical characterization, sacrificing imaging quality, and consequently losing valuable information about the fine microstructure. In this context, this work aims to establish a robust correlation between nanostructure and mechanical properties at the nanoscale of a semi-crystalline polymer using both conventional AFM imaging (tip radius ~5 "nm") and PeakForce™ QNM™ (tip radius ~125 "nm"). Initial results demonstrate a strong correlation between elastic modulus and lamellar organization, with modulus values varying significantly depending on lamellae organization and orientation. More generally, this work aims to propose a reliable mechanical characterization method for semi-crystalline polymers.

14:30 "Effect of electron irradiation on silicate glasses plasticity during in-situ micromechanical tests"

Matthieu Bourguignon[1], Gustavo Rosales-Sosa[3], Etienne Barthel[1], Yoshinari Kato[3], Sergio Sao-Jao[2] and Guillaume Kermouche[2]

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Oxide glasses are the archetype of brittle amorphous materials, but at the micron scale, they can undergo plastic flow, and exhibit a certain ductility. However, there seems to be a connection between this microscopic plasticity and macroscopic brittleness, requiring a better understanding of the specifics of this local plastic flow. In order to highlight this plasticity, microcompression and relaxation tests were conducted on pure silica, aluminoborosilicate glass, and soda-lime silicate glass. These tests indicated that electron irradiation tends to influence the mechanical behaviour of these materials by promoting viscoplastic flow. A time-irradiation equivalence (or strain rate-irradiation equivalence), similar to the well-known time-temperature equivalence in polymers, was established, along with corresponding master curves. The current density sensitivity could be employed to explore the viscoplastic behaviour of silicate glasses at room temperature across six decades of strain rates. Depending on the electron beam and strain rate conditions, it may result in varied compressed-pillar geometries displaying cracks or not. We finally attempt to correlate the origin of these contrasts between glasses with the impact of irradiation on their structure.

14:45 "Novel approach to wear modelling in the nano-scale for molybdenum-based lubricant coating systems"

Bernd-Arno Behrens[1], Gerhard Poll[2], Kai Möhwald[3], Florian Pape[2], Dennis Konopka[2], Kai Brunotte[1], Hendrik Wester[1] and Norman Mohnfeld[1,*]

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Due to the ongoing global economic and environmental shifts, there is a growing demand for innovative and eco-friendly technologies. One solution to address this problem is the use of solid lubricants in rolling contacts to reduce dependence on environmentally harmful greases and oils. This research focuses on developing a solid lubricant system with regenerative properties. The layered system comprises a molybdenum (Mo) reservoir and a top layer of molybdenum trioxide (MoO_3). It is expected that Mo reacts with atmospheric oxygen after wear of the surface and forms new oxides. Measuring the wear volume of thin layers with a thickness between 0.5 to 2 µm on a microscopic level is challenging. Therefore, the study initiates wear behaviour analysis on the nano level. Single Mo and MoO_3 coatings, produced through physical vapor deposition (PVD), are precisely characterised through nano-testing. The primary focus is to determine the wear volume of individual coatings using a newly devised method that considers the initial surface topology. Nano-wear tests, featuring various wear paths and normal forces, are conducted and measured using in situ scanning probe microscopy (SPM). Based on the characteristic values the wear coefficient of the wear model according to Sarkar was calculated. Finally, the developed wear model is validated through additional wear tests on the respective monolayers.

15:00 "FEM study of the validity domain of two analytical nanoindentation models for thin film elastic modulus extraction"

Marina Melo de Lima[1,2], Vincent Mandrillon[2], Laurent-Luc Chapelon[1] and Olivier Lebaigue[3]

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Nanoindentation is widely used for determining the elastic properties of thin films deposited on a substrate. However, the raw measured elastic Young's modulus is a composite film/substrate modulus. Therefore, a common method consists in indenting to less than 10% of the film thickness and considering elastic properties as those of the film alone. However, this method is inadequate for most cases due to the extent of elastic deformation that can reach the substrate and because of tip blunting complexifying ultra low depth indentation analysis. Several studies develop models to obtain the film Young's modulus considering the substrate contribution [1-5]. In this work, a finite element parametric study is performed to identify the validity domain of two recognized models [4] [5] depending on the mismatch of elastic properties between the film and substrate (Efilm/Esubstrate) and on the ratio of the contact radius to the film thickness (rc/tf). The study is performed in the 10-3 < Efilm/Esubstrate < 103 and 10-2 < rc/tf < 102 adimensional parameters ranges. Maps showing results of relative error in Young's modulus retrieval are presented and will help evaluate the confidence of the chosen model.

Talks — Session II

15:45 "Structure-Property Relationships of 3D/4D Printed Microstructures"

Clara Vazquez-Martel[1,2*], Samantha O. Catt[1,2], and Eva Blasco[1,2]

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Additive Manufacturing (AM), also known as 3D printing, has gained much attention during the last years by becoming a promising fabrication tool, which has rapidly influenced not only academia, but also industry. Technologies such as two-photon laser printing (2PLP), allowing precise printing at micro and nanoscales, hold promise for diverse applications from optics and sensing to biomedicine and soft robotics. Although great progress has been made in achieving high resolution, the development of new functional materials is crucial for the successful integration of these technologies into real-world applications. For this purpose, a comprehensive understanding of the behaviour and properties of the printed materials is mandatory. These properties depend not only on the chemical structure or formulation of the material used, but also on a multitude of 3D printing process parameters. In this context, the analysis of structure-property relationships is crucial to design new functional materials for 2PLP. In contrast to the macroscale, mechanical characterization at smaller scales is not straight-forward and only few data for some commercially available materials is available in the literature. The focus of our work relies in developing functional materials for 3D/4D micro- and nanoprinting, including stimuli-responsive inks based on liquid crystalline, shape memory, or conductive materials. Our objective extends to developing methodologies for the comprehensive characterization of 3D printed microstructures, providing insights into structure-mechanical relationships unattainable through conventional methods. Utilizing a nanoindenter for mechanical measurements, this research aims to establish a database for designing and optimizing materials and microstructures across various applications in 3D/4D printing.

16:00 "Investigation of the Deformation Behavior in a CoCrNi Medium Entropy Alloy with Short-Range Order Structure via In-Situ Compression Transmission Electron Microscopy"

Tai Cheng-Ling [1*], Takahito Ohmura [2], II Seiichiro 2, Yang Jer-Ren [1]

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The debated strengthening effects arising from the formation of a short-range order structure (SRO) in an equiatomic CoCrNi medium entropy alloy (MEA) has been investigated. In-situ compression tests were conducted on nanopillars within a TEM (JEOL ARM300F) for both the unaged and aged CoCrNi MEAs. Aging treatment at 600 °C for 5 h was applied to induce the formation of a short-range order structure. In the unaged CoCrNi sample, as compression continued, two slip bands initially formed, followed by an additional two slip bands. However, these slip bands were categorized within the same slip system. The strength burst occurred not due to the sample's softness but because of the space created between the nano-indenter and the nano-pillar when numerous dislocations escaped to the pillar's surface. For the aged CoCrNi sample, when the applied load exceeded the yield strength, multiple slip bands occurred simultaneously, indicating the formation of a uniform SRO in the matrix confined the movement of dislocations, resulting in homogeneous deformation. Additionally, with increased strain, a secondary slip system became active. The confined dislocations readily accumulated, leading to the attainment of the critical stress for the twinning mechanism. In conclusion, the uniform and dense distribution of the short-range order structure in the CoCrNi MEA matrix hinders the movement of dislocations, promoting homogeneous deformation. Furthermore, for the aged CoCrNi sample, the short-range order structure contributes significantly to the strength enhancement, not only through dislocation confinement but also via the formation of deformation twins.

16:15 "Micromechanical characterization of NMC cathodes from Li-ion batteries"

Tobias Sedlatschek[1,*], Malte Schmachtenberg[1], Felix Weber[1], Alexander Bezold[1], and Christoph Broeckmann1

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The understanding of ageing mechanisms in Li-ion battery materials is of great importance to improve the longevity of electrical systems. In this context, mechanical degradation in terms of cracking is one relevant ageing mechanism that must be examined together with the mechanical behavior of battery materials in general. Typical cathodes are composed of active material particles and binder. The active material particles may be single crystals or poly-crystalline agglomerates of very small grains. In this work, we present a complete micromechanical characterization of $LiNi_xCo_yMn_{1-x-y}O_2$ (NMC) cathodes starting on the particle level and ending on the electrode level, i.e. the heterogeneous particle-binder composite. First, an averaged Young's modulus of the NMC particles was determined using targeted nanoindentation. Then, the deformation behavior of individual grains in single-crystalline material was further examined in micropillar compression tests which were followed by micro tensile tests in poly-crystalline material to characterize the interface behavior at grain boundaries. The real cyclic load during electrochemical charge-discharge cycles in batteries was reproduced in micro cyclic tests which allow for the local observation of the material degradation during repeated loading. Finally, the transition to the electrode level was achieved through mesoscopic compression tests of the particle-binder composite under dynamic and cyclic loads and the analysis of the deformation behavior of the electrode. The acquired information allows for an experiment-based understanding of ageing mechanisms and is furthermore highly relevant for the development of reliable physics-based simulation models on the particle level.

16:30 "In-situ XRM study of fracture behaviour of on-chip interconnect stacks with complementary FEM analysis"

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

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Semiconductor industry is continuing the downsizing of on-chip interconnect stack dimensions for both performance and economic considerations. This trend poses significant challenges for the design of crack stop structures within the back end of line (BEoL) stack. On one hand, these protective structures must efficiently stop microcracks from propagating into the interconnect stack, and on the other hand, they must maintain a minimal footprint. To assess the performance of crack stop structures, micromechanical experiments in combination with suitable imaging techniques are required. In this way, a targeted fracture initiation with suitable visualization of the fracture behaviour is achieved. Therefore, a micromechanical in-situ setup was integrated into an X-ray microscope (ZEISS Xradia 800 Ultra). This experimental setup enables high resolution imaging of the 3D-patterned sample structures and occurring defects with a resolution of up to 50 nm. Continuous imaging allows seamless tracking of microcrack propagation during in-situ testing. Fracture is introduced into the BEoL stack by actuating the tailored specimen geometry, where tensile load is applied through a lever mechanism. Additional finite element modeling (FEM) of the sample geometry provides deeper insights into the acting stress and strain fields during loading and helps understanding the observed fracture behavior. This multidisciplinary approach combines experimental techniques, in-situ visualization, and computational modeling to investigate the performance of crack stop structures and gain deeper understanding of the fracture mechanics in on-chip interconnect stack structure.

Talks — Session III

17:00 "New geometry for shear properties measurement and high strain rates testing at micro-scale"

Benedicte ADOGOU[1*], Gaylord Guillonneau[2], Guillaume Kermouche[1], Sergio Sao Joao[1]

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A new method to measure micromechanical properties of materials is developed. This technique was inspired from a macroscopic test named "Shear Compression Specimen", first developed by Dorogoy et al. [1]. It consists of compressing a cylindrical pillar, with two grooves inclined at 45° from the longitudinal direction. The groves are machined at mid-height of the cylinder using FIB technology. This results in a pillar with an inclined gauge. This pillar named "Microshear Compression Specimen" (MCS) can easily be compressed using standard in-situ microindenter device. The technique has first been applied on fused silica. Experimental tests and numerical simulations revealed that the deformation in the gauge is mainly shear, that the shear strain is localized in the pillar's gauge and that the strain is uniform along the gauge. This technique can then offer several advantages: first the ability to probe the shear properties of surfaces and by the fact of localization strain rate demultiplication. This last point has been demonstrated on amorphous selenium were using MCS allowed to reach higher strain rate than what was achievable by simple cylindrical pillars.

[1] A. Dorogoy, D. Rittel, A. Godinger, Exp Mech 55, p. 1627 6 1639, 2015.

17:15 "A nanoindentation study on the mechanical behavior of the cementum-dentine junction in wild boar mandibular molars"

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Cementum-dentine junction (CDJ) is an Interzone connecting two bony tissues in the roots of mammalian teeth namely, cementum and dentine. (CDJ) endures millions of mastication cycles with almost no clinical failure, making it an excellent model for bio-inspired Interzone. We employed nano-dynamic mechanical analysis (nanoDMA) and quasi-static nanoindentation techniques to evaluate the viscoelastic behavior of the (CDJ). A Hysitron TI950 nano-indenter was utilized for testing. To maintain the samples wet during testing, a Berkovich fluid cell tip was used, allowing immersion of the samples in liquid. 2 mm thick slices were cut parallel to the bucco-lingual plane from the second molars of S. scrofa wild boars and subsequently polished. To exclude any potential chemical changes or influence of polymer-filled pores, specimens were not infiltrated with polymer. Moreover, a group of samples were dried for 1 hour at 125°C for comparison with the wet samples. In nanoDMA measurements, average values of indents at the same distances from the CDJ revealed higher storage and lower loss moduli in the dry state compared to the wet state. The storage modulus, representing linear elastic behavior, was minimally affected by frequency, whereas the loss modulus, indicative of viscoelastic deformation components, exhibited an increase with decreasing frequency. The quasi-static analysis on dry samples showed a relatively high elastic modulus at the CDJ region compared to the neighboring regions, followed by a decrease in neighboring areas. It then increased on the dentine side and continued to decrease on the cementum side. The endurance of the CDJ may be attributed to the mismatch of elastic moduli around the interzone.

17:30 "Combining XRD and nanoindentation to characterize mechanical properties evolution of flax cell walls during controlled heat treatment"

C. Caër*[1], E. Guillou2,3, G. Le Saout[4], L. Dumazert5, A. Beigbeder[3], P. Ouagne[6], J.Beaugrand[7], A. Bourmaud[2], N. Le Moigne[5]

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Over the past decades, a tremendous interest has been developed concerning plant fibre used to reinforce composite materials as these fibers show comparable mechanical properties and higher ecological benefits than synthetic fibres such as glass fibres. Environmental concerns are currently driving interest in the replacement of thermoset matrices by thermoplastic polymers and pave the way for recycling and composting as end-of-life routes for plant fibre composites. However, using thermoplastic polymers as a matrix involves long processing cycles at high temperature, which may negatively affect both structure and mechanical properties of plant fibres. In this work, structural evolution and mechanical behaviour of flax fibre cell walls were dynamically monitored in-situ by X-ray diffraction and nanoindentation from ambient to 230°C. A drop in local mechanical performance of flax cell walls, associated with structural evolution at different scales and changes in the biochemical composition, were noticed while increasing temperature. This work, proposing for the first time an in-situ investigation of the dynamic evolution in temperature of the flax cell wall properties, also highlighted the reversible behaviour of their crystalline structure and local mechanical properties, after cooling to room temperature.

17:50 "TI990 - the next generation nanoindenter"

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Bruker's next-generation Hysitron TI 990 TriboIndenter® sets new standards for performance, flexibility, and usability in nanomechanical and nanotribological characterization. A comprehensive advancement of Bruker's industry-leading TriboIndenter platform, every aspect of TI 990's measurement and analysis process features updated technologies designed to remove the normal limitations of nanoindenter systems. As such, this system features the most measurement modes available and delivers high-precision measurements in the broadest range of laboratory environments.

Keynote Lecture

19:45 "Combining nanomechanical testing with AFM and TEM to study small scale plasticity: application to MAX phases"

Christophe TROMAS[1,*], Salomé PARENT[1], Hadi BASOUN[1] and Anne JOULAIN[1]

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Nanomechanical testing, such as nanoindentation, or micropillar compression, is becoming a common technique for probing the mechanical properties of materials at small scale. Beyond measurement, this is also a means of inducing local plasticity in its earlier stages. Under these conditions, it is possible to investigate the elementary mechanisms of plastic deformation in terms of individual dislocations. The combination of different observation techniques such as Transmission Electron Microscopy (TEM) to characterize dislocations or twinning in the volume, and Atomic Force Microscopy (AFM) to observe the traces left on the surface by the emergence of these defects enables complete 3D characterization of these deformation microstructures. In addition, Automated Crystal Orientation Microscopy (ACOM) (in SEM or TEM), can provide crystallographic misorientation maps of the deformed zone. In this presentation, we will apply this approach to study the small-scale plasticity of the MAX phase Cr_2AlC. MAX phase are nanolamellar materials with a hexagonal crystallographic structure. Their plasticity was known to involve basal slip. In this study, we show that deformation twinning is a deformation mechanism that needs to be taken into account in this materials as well as cross slip in prismatic and pyramidal planes.

Abstracts - Oral Presentations

Day 2: Wednesday, March 20th

Talks — Session IV

Invited Talk

09:15 "Spherical Nanoindentation Testing – a promising way to measure localized flow curves"

Verena Maier-Kiener[1]

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In recent years, micromechanical testing techniques based or derived from nanoindentation experiments became a versatile tool for testing local mechanical properties, providing access to properties way beyond hardness and modulus. While standard methods can be easily used for conventional mechanical properties analyses, also a high throughput screening of various materials is conveniently achievable. Moreover, smart adjustments of standard nanoindentation protocols opened an even wider range of applications. For example, it was possible to directly probe dominating thermally activated deformation processes in materials by applying abrupt strain-rate changes within the indentation process. A further appealing idea to material scientists is to characterize the flow behavior of materials with minimal experimental effort while guaranteeing highly reliable results. Thereby nanoindentation has been recently proven to be one possible candidate technique to achieve this objective. Although established as standard method to extract hardness and Young's modulus, the technique is not yet fully exploited regarding the determination of localized flow curves, since understanding the correlations between mechanical properties obtained by spherical indentation experiments and uniaxial data is extremely challenging. To correctly account for tip imperfections, a calibration procedures originating from fundamental geometrical considerations is applied. This sets the foundation for strain-rate controlled experiments and allows an experimental evaluation of the constraint factor in consideration of the mechanical properties and induced strain, which enables the extraction of reliable flow curves and investigation of local workharding behavior. In addition to these advanced testing methods, setups can be further modified in order to probe in-operando materials deformation behavior under ambient, but more importantly under non-ambient conditions, such as temperature ranges from -150 °C to ~ 1000 °C and under electrochemical conditions. Especially the latter allows studying hydrogen materials-interactions and hydrogen embrittlement. This talk will focus on the wide range of possibilities to investigate by micro- and nanomechanical testing methods and their correlation and implementations with respect to computer driven models, as well as to the newly started COST action 21121 MecaNano (European Network for the Mechanics of Matter at the Nano-Scale), which aims to bring together different research areas of micro- and nanomechanical testing, nanomechanical simulations as well as data driven machine learning approaches.

09:45 "Inverse method to determine cyclic properties of materials by combining cyclic indentation and numerical simulation"

Hafiz Muhammad Sajjad [1,*], Thomas Chudoba [2] and Alexander Hartmaier [1]

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Depth-sensing indentations are very convenient experiments to determine mechanical properties (i.e. Young's modulus and hardness) ranging from thin films to bulk materials. In this study, we present a hybrid method to determine material parameters for cyclic plasticity and time-dependent viscoplasticity from cyclic indentation by using simulation-based inverse methods. In order to extract material properties from cyclic indentation data, we combine cyclic indentation experiments and finite-element simulations for a spherical indenter. During the inverse analysis, an iterative optimization process is executed to determine the best fitting material parameters for the simulation by minimizing the difference between experimental and simulated indentation curves. The parameters obtained in this way are then used to predict the uniaxial cyclic stress-strain hysteresis which provide an independent way to validate the result of the inverse parameter identification. This method has been successfully applied to martensitic steel. Finally, the method has been extended to include a holding stage by using a combined creep/plasticity model as constitutive rule for the finite-element simulations.

10:05 "Time-dependent mechanical behavior of semicrystalline polymers by multi-scale characterization"

M. Venin[1], P. Goleo[1], H. Tocqueville[1], and J. Pepin[1,*]
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Semicrystalline polymers may display a high degree of heterogeneity making it difficult to describe their mechanical behavior. Among other factors, the microstructure developed during process can lead to a gradient of properties. Thus, a multi-scale characterization of the mechanical response becomes necessary and instrumented indentation proves to be the most appropriate tool. An injected semi-crystalline polymer has been characterized and indentation allows to highlight the skin/core effect for the elastic properties arising for this process. Nonetheless, one of the main difficulties lies in the viscous nature of polymeric materials which can be probe by two complementary approaches: static nanoindentation (quite commonly used) and dynamic nanoindentation. The second part of this work is to characterize the viscoelastic properties of semi-crystalline polymers at different scales, building the master curve from both DMA and nanoDMA results taking into account the microstructure gradient. To that purpose, experiments were performed at temperatures between room temperature and 120°C varying the frequency over the range 0.3 – 100 Hz at both macro and micro scales. The construction of a master curve, using the Time Temperature Superposition principle, allows to determine the apparent activation energy linked to the relaxation process of this material.

Invited Talk

10:25 "New Insights into the Statistical Nanoindentation Methodology"

Dr. Esteban Broitman[1,*]

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The principle of statistical nanoindentation proposed ca. 2006 is based on performing a relatively large number (many hundreds/few thousands) of single indentation tests in a grid and analyzing the indentation elastic modulus and hardness with statistical methods [1]. Many authors have claimed that this method can be used to study composite materials, showing that mechanical properties, number, and volume of the different composite phases could eventually be deduced and predicted by only using the nanoindentation technique. In this presentation, we first review the previous work done in statistical nanoindentation by different researchers, highlighting the main problems that have been encountered and possible proposed solutions. In the second part, we study and report the statistical nanoindentation of three composite model samples, in the form of a soft Al2124 matrix embedded with hard SiC particles [2]. We propose a novel heuristic wavelet technique to filter the measurement noise from the raw nanoindentation data as an attempt to obtain a more robust statistical nanoindentation methodology. Furthermore, a Finite Elements modeling will be used to analyze the response of the nanoindenter regarding the position of the hard particles [3]. Our modeling will show many mistakes made by authors in previous publications. Finally, we will introduce results on bearing steels. Hardness histograms generated by Statistical Nanoindentation will demonstrate unique characteristics (fingerprints) for different analyzed steels [4].

[1] E. Broitman, "Indentation hardness measurements at macro-, micro-, and nanoscale: a critical overview" Tribology Letters 65 (1), (2017) 23.
[2] E. Broitman et al, "Study of Al2124-SiC nanocomposites by an improved statistical nanoindentation methodology" J. Vac. Sci. Technol. A 41 (6), (2023) 063210.
[3] M.Y. Sherif, E. Broitman, et al, "The influence of steel microstructure in high-speed high-load bearing applications" Mat. Sci. Technol. 37 (17), (2021) 1370-1385.
[4] E. Broitman, et al, "Microstructural Analysis of Bearing Steels by a Statistical Nanoindentation Technique" Bearing World Journal 5, (2020) 47.

10:55 "Strain Rate Dependent Crystal Plasticity Parameter Optimization for CPFEM Nanoindentation Simulations in Single Crystals"

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This research project aims to optimize the crystal plasticity finite element method (CPFEM) parameters for nanoindentation simulations on single crystal materials. The core objective is to develop and validate an automated approach for parameter optimization, focusing on the simulation of single-grain nanoindentation and its application to various testing scenarios. The methodology includes a parameter optimization for the Abaqus software suite, followed by the implementation of automatic control of Abaqus coupled with CPFEM simulations on the CSC platform. The program-driven optimization will specifically address the build-up and validation of approaches for a single-grain conical indenter and a multi-grain cube corner indenter, with an extension to incorporate strain rate-dependent parameters. This study will contribute to the advancement of material science research by providing a systematic approach to calibrating CPFEM models for more accurate and reliable nanoindentation simulations. This study will contribute to the advancement of material science research by providing a systematic approach to calibrating CPFEM models for more accurate and reliable nanoindentation simulations.

11:40 "New Leads on the Densification Profile Underneath Nano Indentation Imprint in Silica Glass by Means of a Chemical Dissolution Technique"

Jean-Pierre Guin[1(*)], Vincent Keryvin[2], Ludovic Charleux[3], Kun. Han[1], Jean-Christophe Sangleboeuf[1], Michael J. Ferry[4]

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The region of permanent densification beneath Berkovich nano-indentation imprint in silica glass is investigated using the chemical probe technique [1]. The later relies on the measurement of local chemical reactivity variations resulting from the structural modifications of the densified glass in the plastic zone by successive atomic force microscopy imaging of the same residual imprint through dissolution steps. The use of the similitude regime in sharp indentation testing allows one to record reliable data with a good spatial resolution that makes it possible to deal with low loads (typically below 10 mN) and, more importantly, crack-free residual imprints. The densified zone dissolves much quicker than the non densified regions. The analysis of the results, along the vertical axis are conducted within the geometrical similar framework [2, 3] which makes it load independent but also re-scalable to any load for comparison if needed. Results indicate that the densification zone, along this axis is made of 2 zones: the first one just beneath the residual imprint is rather homogeneous in terms of dissolution rate thus in terms of densification ratio and the second one is a rather steep transition to the non densified zone. A reactivity model linking densification ratio (through structural modifications) [4, 5] to dissolution rate has been developed [6]. It allows: on the one hand the validation of possible densification profiles such as two parameters sigmoidal functions for which the sole adjusting parameters to match the experimental points is the width of the transition zone as its position is provided by the experimental results; and on the other hand to gather all the densification profiles available in the literature wether they originate from Raman spectroscopy mapping (loads in the 1 N range) our from numerical modeling and compare them to the chemical probe results either in terms of imprint depth evolution through dissolution steps or in terms of densification profile all of this being treated within the similar geometry frame work of pyramidal indentation as, for example, described in references [2, 3] . Agreements and disagreements with data from the literature are discussed in terms of possible densification profiles, geometrical similarity as well as Raman confocal probe drawbacks. Limitations of both the chemical probe technique and Raman spectroscopy mapping are addressed and perspectives are drafted out.

1. Y.-F. Niu, K. Han, J.-P. Guin, Locally enhanced dissolution rate as a probe for nanocontact-induced densification in oxide glasses., Langmuir. 28 (2012) 10733–40.
2. Cheng, Y. T. & Cheng, C. M. Scaling, dimensional analysis, and indentation measurements. Mater. Sci. Eng. R Reports 44, 91–149 (2004).
3. D. Tabor. The physical meaning of indentation and scratch hardness. Br. J. Appl. Phys. 7, 159 (1956).
4. Hehlen, B. Inter-tetrahedra bond angle of permanently densified silicas extracted from their Raman spectra. J. Phys. Condens. Matter 22, 25401 (2010).
5. Sonneville, C. et al. Polyamorphic transitions in silica glass. J. Non. Cryst. Solids 382, 133–136 (2013).
6. Bunker, B. C., Haaland, D. M., Michalske, T. A. & Smith, W. L. Kinetics of dissociative chemisorption on strained edge-shared surface defects on dehydroxylated silica. Surf. Sci. 222, 95–118 (19

12:00 “Exploring chemomechanical weakening in muscovite via liquid nanoindentation”

Frank W. DelRio[1,*], William M. Mook[1], Anastasia G. Ilgen[1], Katherine L. Jungjohann[2], Wendelin Wright[3], Jordan Sickle[4], and Karin A. Dahmen[4]

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In this talk, chemomechanical weakening of layered phyllosilicate muscovite mica was studied as a function of chemical environment via in-situ liquid-nanoindentation under four conditions (dry, deionized (DI) water, and two NaCl solutions of different pH). While traditional indentation analyses of layered materials with extreme mechanical anisotropy have been limited due to displacement bursts (pop-ins), here the bursts were used as proxies for delamination, fracture, and spalling events. Since displacement bursts during an indent represent a stochastic process, 120 indents were conducted for each condition to generate statistically-significant amounts of data. In total, over 9000 bursts were assessed using a load-displacement threshold criterion, classifying this as a high-throughput nanoscale fracture technique. For each burst, initiation load, initiation displacement, plastic zone volume at initiation, and energy dissipation were analyzed. A power-law relationship between the burst load and burst energy was noted which separated the bursts into two continuous distributions: (1) bursts due only to the mechanics of the indent and (2) bursts due to both the mechanics of the indent and the environment. By using a cumulative probability distribution, it was found that the NaCl solutions decreased the minimum plastic zone volume necessary to initiate a displacement burst by an order of magnitude relative to the dry condition. Finally, the underlying mechanisms explaining the trends in initiation volume as a function of environment were discussed, with a focus on the degradation processes via chemical attack and cation exchange. SNL is managed and operated by NTESS under DOE NNSA contract DE-NA0003525.

12:20 ”Irreversible evolution of dislocation pile-ups during cyclic microcantilever bending”

Szilvia Kalácska[1*], Dávid Ugi[2], Kolja Zoller[3], Kolos Lukács[2], Zsolt Fogarassy[4], István Groma[2], Katrin Schulz[3] and Péter D. Ispánovity[2]

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In metals geometrically necessary dislocations (GNDs) are generated primarily to accommodate strain gradients and they play a key role in the Bauschinger-effect, strain hardening, micron-scale size effects and fatigue. During bending large strain gradients naturally emerge which makes this deformation mode exceptionally suitable to study the evolution of GNDs. Here we present bi-directional bending experiment of a Cu single crystalline microcantilever with in situ characterization of the dislocation microstructure in terms of high-resolution electron backscatter diffraction (HR-EBSD). The experiments are complemented with dislocation density modelling to provide physical understanding of the collective dislocation phenomena. We find that dislocation pile-ups form around the neutral zone during initial bending, however, these do not dissolve upon reversed loading, rather they contribute to the development of a much more complex GND dominated microstructure. This irreversible process is analysed in detail in terms of the involved Burgers vectors and slip systems. We conclude that at this scale the most dominant role in the Bauschinger-effect and corresponding strain hardening is played by short-range dislocation interactions. The in-depth understanding of these phenomena will aid the design of microscopic metallic components with increased performance and reliability [1].

[1] D. Ugi et al., Materials & Design (2024) DOI: 10.1016/j.matdes.2024.112682

Invited Talk

12:40 "Quantification of electron-irradiation creep in amorphous olivine and amorphous silica from In situ TEM and SEM nanomechanical testing"

Guillaume Kermouche[1,*], Paul Baral[1], Sergio Sao-Joao[1], Andrey Orekhov[2], Dominique Schryvers[2], Patrick Cordier[3]; Mickael Coulombier[4], Thomas Pardoen[4], Hosni Idrissi[4], Etienne Barthel[5], Mathieu Bourguignon[5], Gustavo Rosales[6]

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Oxide glasses are the archetype of brittle materials, but they are known to exhibit large ductility when loaded at a submicronic scale. For instance, micron-sized silica pillars were successfully compressed up to large plastic strain without failure. Amorphous olivine exhibits large tensile ductility at the size of a few hundreds of nm. Most of these results were obtained using in situ nanomechanical testing in SEM or TEM to allow imaging the deformation mechanisms. However it was observed that electron irradiation sometimes played a role on the ability of these materials to sustain such large plastic strain, which might lead to some erroneous conclusions. In this paper we propose two nanomechanical testing methodologies to quantify the effect of electron-irradiation. The first one consists in using multiple "beam off – beam on" cycles to enhance electron-irradiation creep during tensile deformation applied through the push-to pull device. The use of a simple uniaxial elastic-viscoplastic formalism accounting of the push-to-pull stiffness allows the quantification of yield parameters such as the strain-rate sensitivity or the activation volume as a function of irradiation parameters. Interestingly, it is shown that this simple uniaxial model is very helpful to analyse load-controlled push-to-pull results. The second method is the micropillar relaxation testing. It consists in applying a prescribed compression strain at a given strain rate on a micro-pillar and then let the material relax under electron-irradiation. This kind of test allows the identification of a time-irradiation equivalence similar to the well-known time-temperature equivalence in glassy materials.

Talks — Session VI

Invited Talk

14:30 "In-situ TEM straining experiments to probe dislocation mechanisms"

F. Mompiou[1*], Marc Legros[1], Daniel Caillard[1]
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Observing deformation at the pertinent time and length scale is a goal that material scientists dream of. With the advance of fabrication techniques, micromechanical testing, and imaging techniques, in the last 20 years, the community has been able to address plastic deformation mechanisms based on nanometric in-situ experiments. However, despite these progresses, conclusions drawn from such experiments has suffered from critics because of possible lack of reproducibility, caveats and artifacts. In this talk, we would like to present a critical analysis of in-situ tensile tests performed on electropolished samples based on data accumulate over the years at CEMES. The "straining technique" was the first to emerge in earlier days of electron microscopy and despite being more confidential in the recent years, it has continued to produce valuable results. It allows to observe dislocation dynamics in wide area (several micron square) using conventional bright/dark field TEM imaging and electron diffraction, suitable to allow 1) observations of dislocations at the pertinent length scale (grain size) and time scale (few tens of ms) and 2) fast reciprocal and direct spaces analyses for orientation and 3D information retrieval. Instrumental set-ups make experiments available in a wide range of temperatures, enabling the investigation of thermally activated mechanisms. Contrary to actual nano-indentor set-ups, such straining holders cannot give access to the applied stress. However, observations of curved dislocations provide rich information on local stresses. Here, we will give few examples of observations in high entropy alloys, dilute solid solution alloys and pure metals, that allow to discuss plasticity mechanisms in particular with respect to macroscopic mechanical tests and discuss possible artefacts and caveats.

Ref: Legros, M., Mompiou, F., Caillard, D., 2024. Observing deformation in situ. Nat. Mater. 23, 20–22. https://doi.org/10.1038/s41563-023-01739-2

15:00 "A novel combination of lab-on-chip testing, nanoscale DIC, and ACOM-TEM for unraveling plasticity mechanisms in UFG freestanding metal thin films"

Ankush Kashiwar[1,2,*], Paul Baral[1,3], Michaël Coulombier[1], Laurent Delannay[1], Jean Pierre Raskin[4], Thomas Pardoen[1,5], Hosni Idrissi[1,2]

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Nanocrystalline (nc) and ultrafine-grained (UFG) metals with grain sizes in submicron scales exhibit outstanding mechanical strength and fatigue properties compared to their coarse-grained counterparts. Thin films of nc or UFG metals are promising candidates for various structural and functional applications. However, their applicability is often restricted by the limited ductility exhibited by them due to low dislocation storage capacity within small-sized grains. Besides dislocation-based processes, the complex interplay of grain boundary (GB) mechanisms is well-known to control their plasticity. In the present work, elementary plasticity mechanisms are studied in UFG Al freestanding thin films with exceptional ductility (up to 17%). The mechanisms of plastic deformation are unraveled using a new combination of nanomechanical lab-on-chip testing with nano-scale digital image correlation (nano-DIC) followed by a correlative investigation by automated crystal orientation mapping in TEM (ACOM-TEM). nano-DIC allows for high-resolution quantification of in-plane shear strain along with the rigid body rotation of some clusters of grains across the specimen. The correlation of nano-DIC and ACOM-TEM measurements allows to distinguish intragranular and GB-mediated plasticity and offers a quantitative evaluation of the shear displacement at GBs. These results were further linked with the GB character, orientation, and defects in the deformed specimen. The combination of nano-DIC and ACOM-TEM brings new insight into the relation between local strain and microstructural features in nc FCC films based on a statistically representative study.

15:20 “Automated co-localized imaging and analysis for high-throughput in-situ nanoindentation – Hysitron PI 89 Auto SEM PicoIndenter”

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Bruker’s PI 89 Auto introduces full automation to the market-leading Hysitron PI 89 SEM PicoIndenter. This innovative tool streamlines correlative structure-property analysis by automatically positioning the Rotation and Tilt Stage (R/T stage) for indentation after SEM imaging and EBSD/EDS mapping. The integration of the TriboScan Auto software with the R/T stage enables the PI 89 Auto to achieve unprecedented high-throughput testing with exceptional precision and control. Key features of the PI 89 Auto include seamless transitions between nanoindentation, standard SEM imaging, and EBSD/EDS analysis, co-localized acquisition of quantitative in-situ mechanical data at user-defined regions of interest, and increased throughput without compromising precision or accuracy.

15:40 “Nanomechanical testing of argyrodite particles for solid battery electrolyte applications”

Johannes Ast[1*], Quentin Dupuis[1], Benoit Mathieu[1], Sébastien Liatard[1], Thibaut Gutel[1]

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The present study deals with the determination of micro-mechanical properties of irregular shaped argyrodite particles. Nanoindentation experiments were carried out using a scanning electron microscope (SEM). The main interest was the feasibility of such experimental tests on air-sensitive materials, their reproducibility, as well as the determination of mechanical properties such as Young’s modulus, shear modulus, yield strength and hardness. For that purpose different indentation techniques were applied consisting of flat punch and Berkovich indentation tips. Results show a significant plastic deformation behavior and Young’s moduli, which lie in the expected range for such materials. The experiments are accompanied by indentation simulations to better understand the deformation process of the particles during the powder compaction process.

16:00 “Understanding the micromechanical deformation of anodes, cathodes and solid electrolytes in solid state lithium ion batteries”

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An all-solid-state battery would revolutionise the electric vehicles of the future. The successful implementation of an alkali metal negative electrode and the replacement of the flammable organic liquid electrolytes, currently used in Li-ion batteries, with a solid would increase the range of the battery, reduce recharging time and address the safety concerns. Current efforts to commercialise such batteries worldwide are failing and will continue to fail until we understand the fundamental failure processes taking place in these devices, which are inherently driven by mechanical failure on the nano- and micro- scale. In this talk I will show our development of both testing methodologies and understanding of materials behavior in key materials for solid state batteries. This will include the 1) use of in-situ SEM nanoindentation to understanding, the time dependent and anisotropic elastic and plastic deformation of metallic lithium and lithium based alloys for use as anodes in SSBs. 2) In situ microcantilever fracture testing of Li_6PS_5Cl and Li_6PS_5Cl based composite solid electrolytes under a range of processing conditions to show the often understated (in the battery community) effect of processing on battery performance and 3) ex-situ developments of de-adhesion testing use nano-scratch for understanding failure in cathode thin films.

16:50 "Enriching nanoindentation with in-situ electrical measurements and SEM observations"

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A nanoindentation set-up coupled to electrical measurements and integrated into an SEM will be presented. Its application to the understanding of the interplay between mechanical and electrical behaviors will then be discussed. First, it will be shown how electrical measurements can be used to monitor the contact area between the tip and the sample during indentation. The case of ideal materials (oxide-free) or real materials (with a passivating surface oxide) will be considered. The in-situ SEM configuration is used for the precise positioning of indents on a complex system (precision ~100nm). The local mechanical properties will then be extracted accurately at a micrometer-scale. Then, the interplay of mechanical and electrical behaviors of dielectric thin films will be addressed. First, we will show how a mechanical stress can modify the electrical conduction mechanism in an ultra-low-k dielectric film. Counterintuitive observations will be fully explained numerically (by FEM analysis) by correcting the Poole-Frenkel conduction law with a strain-dependent factor. A threshold strain is identified as the keystone linking this strain-dependent conduction to the current line distribution within the dielectric. Second, we will show how an electrical stress can degrade the mechanical properties of dielectrics. Experiments carried out on various dielectric systems will be described and the mechanical collapse of the film will be explained by the injection of electrical charges into the dielectric. This presentation aims at demonstrating the power of coupling electrical measurements to nanoindentation, either to process mechanical raw data or to understand the strong mechanical-electrical interplay in materials.

17:10 "Eliciting stable nanoscale fracture in single-crystal silicon"

Frank W. DelRio[1,*], Scott J. Grutzik[1], William M. Mook[1], Sara Dickens[1], Paul G. Kotula[1], Eric Hintsala[2], Douglas Stauffer[2], and Brad L. Boyce[1]

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Silicon microelectromechanical systems are often fabricated via bulk and surface micromachining techniques, and as such, possess a number of flaws of varying scale, geometry, and residual stress state, depending on the fabrication method and mechanical environment. In the presence of a mechanical load, these preexisting flaws become sites for stress concentrations, thus making fracture toughness a key metric for component reliability. Historically, fracture toughness has been measured via ex-situ methods such as scratch, indentation, bulge, and stressed overlayer tests. More recently, toughness has been evaluated in-situ in a scanning electron microscope (SEM) to visualize the fracture path and extract multiple values from each specimen. In this talk, we demonstrate stable nanoscale fracture in single-crystal silicon using an in-situ wedge-loaded double cantilever beam (DCB) specimen. The fracture toughness KIC was calculated directly from instrumented measurement of force F and displacement d via finite element analysis with frictional corrections (Figure 1). Measurements on multiple test specimens were used to show KIC=0.72±0.07 MPa m1/2 on {111} planes and observe the crack-growth resistance curve in <500 nm increments. The exquisite stability of crack growth, instrumented measurement of material response, and direct visual access to observe nanoscale fracture processes in an ideally brittle material differentiate this approach from prior DCB methods.
SNL is managed and operated by NTESS under DOE NNSA contract DE-NA0003525.

17:30 "In-situ scanning probe microscopic (SPM) Imaging of Cu pumping phenomenon in hybrid bond wafers"

Ali Roshanghias[1], Jaroslaw Kaczynski[1], and Ude Hangen[2]

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Hybrid Bonding is gaining tremendous momentum in the semiconductor industry as it allows for fabricating very high-density interconnects between functional tiers via wafer-to-wafer and die-to-wafer bonding. Hybrid bonding is based on dielectric-dielectric (SiO_2-SiO_2) bonds at room temperature followed by a low-temperature annealing process to establish metal-metal (Cu-Cu) bonds. In this study, the Cu pumping phenomenon as the enabler of hybrid Cu/ SiO_2 bonding was investigated and quantified using in-situ scanning probe microscopic (SPM) imaging at different temperatures up to 400 °C. The irreversible deformation of Cu after cooling back to room temperature was verified. The experimental results showed good agreement with the FEM simulation results. Implementing both empirical data from SPM and the results of a parametric simulation study, Cu pumping at high temperatures was consequently presented as a numerical function of Young's moduli and temperature.

Invited Talk

17:50 "Neural networks capture the deformation of lattice metamaterials"

Brad L. Boyce
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Structural lattice metamaterials, otherwise known as architected or programmable materials, provide a topological pathway to create effective material properties that cannot be matched by monolithic materials. They enable tradeoffs between material properties and density as well as offering unusual material properties such as negative Poisson ratio. Such lattice materials are now achievable with commercial manufacturing tools at both the microscale and macroscale, and new process technologies with further flexibility in both scale and material are emerging rapidly. Complementary capabilities are being developed to experimentally measure and model the properties of these intrinsically multiscale materials. However, explicit direct numerical simulation through finite analysis is both computationally expensive and often does not capture the heterogeneous imperfections that strongly influence the behavior of as-printed lattices. As an alternative, deep learning networks can be trained to provide a reduced order surrogate model of behavior. We demonstrate the utility of a deep convolutional neural network to predict the deformation response based solely on raw images of the as-printed lattices. Such a tool can not only provide powerful screening for product acceptance, but also lend mechanistic insight into the structural features that control deformation behavior. Armed with such new tools, it is now possible to efficiently design novel lattice topologies to optimally satisfy multiple objectives on a Pareto front. This approach provides an alternative to traditional topology optimization, which is typically limited to linear design problems such as elastic response.

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Abstracts - Oral Presentations

Day 3: Thursday, March 21st

Talks — Session VIII

Invited Talk

09:00 “Nanomechanical testing of steels using nanoindentation”

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Modern steel grades and metallic coatings exhibit complex microstructures composed of multiple constituents of sub-micron size. Control over the distribution, size and properties of each micro-phase is essential in defining the final product behaviour for specific applications, and call for nanoscale characterization methods. The advanced characterization of such products is performed in OCAS using the advanced analytical tools present in our microscopy center : scanning and transmission electron microscopy, focused ion beam (FIB) electron back-scattered diffraction (EBSD), electron probe micro-analysis (EPMA) and nanoindentation Recently, a Hysitron TI Premier nano-indentor was installed at OCAS to strengthen our nano-mechanical characterization capabilities on metals and coatings. In addition to quasi-static, dynamic nano-mechanical testing and in-situ topographical imaging, the system features the “Accelerated Property Mapping – XPM” module. The XPM module enables fast quantitative mapping of elastic modulus and hardness at spatial resolution in the nanoscale range. Nano-hardness values of steels were measured x60 times faster with the XPM module than with conventional nanoindentation, thus giving large datasets (~103 indents) and better statistics. In a first step, the linear correlation between average XPM nano-hardness and micro-Vickers hardness of various steels was validated. Then, the link between microstructural constituents and nano-mechanical properties were investigated using high-resolution XPM maps of martensitic steels. Different chemistries and heat treatments were applied to induce changes in the microstructure of steels and evaluate the effect on their nano-hardness.

09:30 “Local mechanical properties of oxide inclusions”

Sandor Lipcsei[*1], Alejandra Slagter [1,2], Jonathan Aristya Setyadji [1,3], David Hernández-Escobar [1], Joris Everaerts [1,4], Léa Deillon [1,5], and Andreas Mortensen[1]

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Oxide inclusions, a by-product of the deoxidation process that must nearly always be conducted during steelmaking, have a deteriorating effect on the mechanical behaviour of steel. Improvements in steelmaking are leading to increased ranges and control of inclusion composition, while at the same time our ability to assess the intrinsic mechanical properties of small objects such as discrete micron-scale second phases in alloys has progressed significantly over the past ten to twenty years. We thus present here an exploration of the link between inclusion composition and local, in-situ, inclusion mechanical properties. Our goal is to identify inclusion compositions that have properties that minimize their negative impact on the mechanical performance of steel. To this end, we use an adaptation of the Oliver-Pharr method to measure, via nanoindentation, the hardness and stiffness of inclusions embedded in a matrix, taking into account the influence that is exerted on the indenter load-deflection signal by the matrix to deconvolute inclusion properties from local nanoindentation measurements conducted on individual inclusions. Using the proposed indentation data analysis procedure, we measure the influence of composition on the stiffness and hardness of inclusions belonging to the $SiO_2 – Al_2O_3$ – CaO and $SiO_2 – Al_2O_3$ – MnO systems.

Acknowledgement: This work was performed under support of the Swiss National Science Foundation, Project No. 200020 215297/1

09:50 "Correlation at the sub-micrometric length scale between surface integrity and microstructure in WC-Co grades"

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Cemented carbides are composite materials widely used in different industry fields within applications involving wear, due to their outstanding wear resistance. The most commonly used are WC-Co grades, for Co wettability with the carbide and adhesion characteristics. The remarkable mechanical integrity of these materials result from a two-fold effectiveness associated with their intrinsic composite character. On the one hand in terms of composite nature and on the other as related to composite assemblage. In particular this presentation is focused on WC-Co hardmetals as reference hard material. A large number of studies have been reported on the mechanical behavior of this composite. However, information on the small-scale mechanical response of these materials as a function of the superficial state is rather scarce. This is particularly true regarding experimental data and analysis on the influence of phase nature, crystal orientation (anisotropy) and interfacial adhesion strength on hardness, deformation and/or damage mechanisms. It is clear that knowledge and understanding of these issues is crucial not only to enhance the performance of hardmetals but also to develop ceramic-metal composites beyond WC-Co systems. In this regard, this work was conducted to find a correlation between the microstructure and mechanical properties at the submicrometric length scale of WC-Co cemented carbides polished by using the DryLyte® technology. The attained mechanical property mapping under different superficial states, presents a clear correlation between local hardness and stiffness with the resulting roughness and also with the superficial stress state induced during the pre-processing process.

10:10 "Friction and roughness at the nanoscale"

Alexia Crespo[1], Lucas Frérot[2], Mark Robbins[2], Denis Mazuyer[1] and Juliette Cayer-Barrioz[1,*]
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As fluid viscosity decreases leading to a smaller surface separation in contact, a growing interest on surface topography has emerged in the recent years. This work focused on the role of roughness, investigating at the nanometric scale both the film formation and the friction, using a molecular tribometer in sphere/plane configuration.

Here we explain, for a frictional system exclusively undergoing structural aging, how the macroscopic friction response emerges from the interplay between the surface roughness and the molecular motion within adsorbed monolayers. The existence of contact junctions and their friction dynamics are studied through coupled experimental and computational approaches. The former provides detailed measurements of how the friction force decays, after the stiction peak, to a steady-state value over a few nanometers of sliding distance, while the latter demonstrates how this memory distance is related to the evolution of the number of cross-surface attractive physical links, within contact junctions, between the molecules adsorbed on the rough surfaces. We also show that roughness is a sufficient condition for the appearance of structural aging. Using a unified model for friction between rough adsorbed monolayers, we show how contact junctions are a key component in structural aging and how the infrajunction molecular motion can control the macroscopic response.

10:30 "Identification of the mechanical and electrical behaviors of a copper oxide layer by resistive-nanoindentation tests"

M. Rusinowicz[1], F. Volpi[1], C. Boujrouf[1], M. Verdier[1], G. Parry[1], M. Braccini[1,*]

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Oxide scale on metal surface is either sought, in the form of a protective passive layer for instance, or needs to be avoided, to limit its interaction with electrical conductivity for example. In both cases, mechanical and conductive behaviors of the oxide layer are key parameters and need to be measured. The resistive-nanoindentation technique appears as an appropriate approach since mechanical and electrical properties of a system can be measured simultaneously, as well as their interplay. In this study, a Cu_2O single layer of 225 nm in thickness was obtained by low-temperature oxidation of an annealed OFHC copper. The system was tested by resistive-nanoindentation consisting in indenting the oxide layer while applying a DC voltage to the metal substrate and measuring the current through the conductive indenter tip. Due to the complexity of the problem, a numerical simulation is required to analyze the interplay between mechanical behavior and electrical response of the system. Indeed, the contact surface between the indenter tip and the material evolves during indentation and depends on the mechanical behavior of the system. Moreover, the densification of the oxide affects the layer thickness and then the local electric field. The elastic modulus of the oxide was thus determined, and its plastic behavior was described using a Drucker-Prager model. The impact of the oxide layer on the plastic behavior of the metal underneath was also demonstrated. Finally, the conduction mechanism in the oxide layer was described by a Poole-Frenkel conductivity law.

Invited Talk
11:20 "Memory effects in soft matter friction: the role of sliding inhomogeneities"

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Unsteady-state frictional situations have long been recognized to involve memory effects. A typical example is the response of a contact when the slip rate is changed suddenly from one value to another greater value: a positive jump in the frictional stress followed by a long-term decay to steady-state is then observed. In order to describe these observations, phenomenological approaches such as the seminal state-and rate model by Rice and Ruina [1] have been introduced where phenomenological state variables account for the fading memory of the contact. However, the underlying physical mechanisms behind state-and-rate friction laws remains largely debated. In this study, we tackle memory effects in friction from the perspective of the transient sliding inhomogeneities which result from the deformation of finite size contact areas during unsteady state sliding. For that purpose, a smooth, single-asperity, contact interface between a deformable rubber and a rigid spherical probe is perturbed by the application of either non rectilinear sliding motions or a velocity step. In the case of non rectilinear motions, we show from measurements of the displacement fields within the contact that stress and strain inhomogeneities keep a memory of the past trajectories. As a consequence of these memory effects, the friction force may no longer be aligned with respect to the sliding trajectory. These observations are adequately accounted for by a friction model which takes into account heterogeneous displacements within the contact area [2]. When a velocity dependence of the frictional stress is incorporated within the model, unsteady state regimes induced by velocity steps are also adequately described. The good agreement between the model and experiments outlines the role of space inhomogeneities in memory effects involved in soft matter friction.

[1] Rice J, Ruina A. Stability of steady frictional slipping. Journal of Applied Mechanics, 1983 50, 343–349.

[2] V.Fazio and V. Acito and F. Amiot and C. Frétigny and A. Chateauminois, Proceedings of the Royal Society A, 2023 A477, 20210559.

11:50 "Fretting behavior of an electrical contact"

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Fretting stress describes a movement of two friction partners against each other with an amplitude smaller than the radius of the contact surface. Fretting stress creates a third body whose properties influence the wear behavior of the contact. A Hysitron TI 950 is equipped with a piezo stage. Additional data acquisition and a synchronization of load function and piezo stage control allow to use the indenter as microfretting tester. A fretting contact pair is analyzed. Hysteresis and contact resistance of an electrical contact are analyzed for different amplitudes. The measured values of contact resistance and hysteresis shape, together with analyses in the SEM, FIB and XPS are used to understand the processes in the fretting contact.

12:10 "Modeling the squeezed-thin film accounting for adsorbed nanometric monolayers"

Denis Mazuyer[1,*], Van-Vuong Lai[2], François Sidoroff and Juliette Cayer-Barrioz[1]

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In order to simulate the squeeze of a confined thin film between two adsorbed nanometric layers on antagonistic solid surfaces, we developed a numerical model based on continuum mechanics using the generalized œdometric Reynolds equations extended to the case of heterogeneous films such as two adsorbed layers separated by a fluid film. This model accounted for the film compressibility and the substrate deformation for a sphere/plane configuration in both static and dynamic situations, under small-amplitude oscillations as used in non-contact dynamic surface forces or AFM measurements for instance. For the static contact resolution, an explicit approach was chosen based on small incremental displacement of the sphere towards the plan. For the dynamic situation, the generalized Reynolds model was assumed still valid with a harmonic solicitation and a complex solution was obtained. Numerical resolution of the governing equations was carried out thanks to Finite-Element-Method in Matlab. Our numerical results were validated thanks to direct confrontation to theoretical and experimental ones. Our model was able to predict such as contact stress or complex stiffness as well as to provide physical insights of the squeeze mechanisms. The application of our numerical modeling to experimental squeeze of low viscosity fluid between fatty acid monolayers allowed one to assess the mechanical properties of the adsorbed nanometric layers and to discuss the molecular organization within the squeezed interface. This method opens new perspectives for measuring viscoelastic properties of adsorbed nanometric monolayers and their evolution under confinement.

12:30 “Tribochemistry of Sulfur Containing Additives on DLC coatings: Contribution of Electronic Spectroscopies”

Aslihan SAYILAN[1,2], Jules GALIPAUD[1,2], Julien FONTAINE[1], Jean-Michel MARTIN[1], Christophe HEAU[3], Lucile JOLY-POTTUZ[2], Karine MASENELLI-VARLOT[2], Maria-Isabel de BARROS-BOUCHET[1]

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Solid and liquid lubrication has a significant role on reducing energy consumption and enhancing the wear behavior of mechanical parts. Diamond-like-carbon(DLC) films are widely used in transportation and wind energy sectors; therefore, it is necessary to investigate their tribological performance in lubricated conditions. They have high chemical inertia under static conditions towards the environment due to their carbon- and hydrogen-based composition. However, in lubricated conditions a “chemical reactor” is activating under severe dynamic contact which is correlated with the breaking of C-C and C-H bonds with shear. This leads the forming of C° free radicals and dangling bonds on contact zone. Also, variety of tribochemical reactions occur with the lubricant molecules at contact asperities. In this research, it is aimed to investigate the reactivity of four different DLC materials: a-C, a-C:H(20), a-C:H(40) and ta-C. Gas phase lubricated tribological experiments are performed in a specific device, “Environmentally Controlled Analytical Tribology platform(ECAT)”, equipped with in situ chemical analyses of surfaces(XPS, Auger, REELS). The tests are carried out in ultra-high vacuum(10-9 mbar) and also by introducing dimethyl sulfide and dimethyl disulfide with increasing pressure to examine the formed tribofilms and the reactivity of different DLC coatings in gaseous lubrication. It is aimed to pay a particular attention on the modification of the chemistry and hybridization state (sp2/sp3) in extreme surface to understand the tribochemical mechanisms. Eventually, this study is targeting to enhance the optimization of multi-functional DLC surfaces by considering their mechanical and chemical properties and to provide better control their friction behavior.

12:50 “The interest of nanoscale tests in the prediction of tribological behavior of dynamic system”

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Tribological behavior of dynamic systems at real scale is often impacted by the local mechanical and physicochemical properties of the surfaces in contact. Nanoscale tests such as nanoindentation and nanoscratch are mainly used to evaluate the mechanical and tribological properties of extreme surfaces, thin films and coatings. Nanoindentation allows the local hardness and elasticity of materials. Nanoindentation is also used to extract the strain-stress curve parameters of different materials. Nanoscratch determines the apparent and local friction coefficients between the indenter and material. It also allows the characterization of the anti-wear properties of materials and the force adhesion of coatings.

Abstracts - Poster Presentations

1. "Multi scale in-situ micromechanical testing of Polymer Core Solder Ball (PCSB) coatings for BGA interconnections"

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In the field of electronic packaging, European directives, constant miniaturization, and the quest for reliability have led microelectronics manufacturers to propose new solutions for their BGA interconnections. Recently, new solder balls made from coated polymers have emerged [1,2]. These balls are obtained using an electroless plating process. The microstructure of these materials differs from bulk materials, as do their mechanical properties. However, bulk material properties are commonly attributed to them. In this study, we focus on the characterization of the mechanical properties of a Cu/Ni-P multilayer coating deposited on a Polymer Core Solder Ball (PCSB). In this coating, the copper is nanocrystalline, while the nickel–phosphorus is amorphous. To understand the influence of these microstructural differences on the mechanical properties of the PCSB, a multiscale study is performed. First, at the microscopic scale, the characterization of the mechanical properties (Young modulus and Yielding strength of two coatings (Cu, Ni-P) is conducted. For this purpose, in-situ micro-mechanical tests (Micro pillar compression and nanoindentation) are carried out. Then, at the mesoscopic scale, the previous properties are validated by applying compressive loading to a PCSB solder ball. A correlation between the numerical and experimental approaches is performed and reveals that the coating properties differ from those of the bulk material [3].

[1] Miettinen J. and al; Stacked 3-D MCP with plastic ball vertical interconnections. Proceedings - Electronic Components and Technology Conference, 2003, 1101–1105.
[2] Shih T. and al; IMC integrity for Sn96.7-Ag3.7 polymer core solder ball in BGA package. Proceedings of Technical Papers - International Microsystems, Packaging, Assembly, and Circuits Technology Conference, IMPACT, 2011, 427–430.
[3] Malkorra I. and al; Multi-scale in-situ micro-mechanical characterization of Polymer Core Solder Ball (PCSB) coatings for BGA interconnections. Microelectronics Reliability 148 (2023) 115135

2. "Obtaining of Bulk Ultrafine Grained Multicomponent FeWAlTiNi Composite Materials by Ball Milling and Explosion Densification Technology"

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There are known several methods for obtaining of ultrafine grained or/and nanostructured Bulk materials in multicomponent systems, basically: hot isostatic pressing (HIP), spark plasma syntheses (SPS), Laser engineering (LE), self-propagating high temperature syntheses (SHS) etc. Conventional technologies do not allow fabricate the bulk ultrafine grained materials in multicomponent systems, because of problems: a) obtaining nanopowders; b) fabrication large scale bulk nanostructured samples. Problems are increase proportionally to the number of components in system. So, development of technologies for nanopowder production and synthesis of large-scale bulk nanocomposites is a challenge. The two-stage method was selected for investigation to obtain bulk nanocomposites in FeWAlTiNi multicomponent system in the presented paper: 1. obtaining multicomponent ultra-disperse mixture by ball milling of precursor composition: iron bases stainless steel powder (X18H15)–W-Al-Ti-Ni with equal mass concentration of components and 2. synthesis of bulk multicomponent nanocomposites by shock densification (SD) of ultrafine powder composition. The percentage of components in precursor mixtures was determined based on thermodynamic analysis and phase diagrams. Planetary ball mill ("Fritsch") was used for nanopowder preparation, with processing times varied in range of 14-56h. In the process of mechanical alloying, the ratio of the total mass of the balls to the mass of powder was 10:1. The particle size and phase state of the powders was monitored by XRD, nano-sizer and scanned electronic microscopes (SEM). The main technological parameters of ball milling processing and SD of powders for fabrication of multicomponent bulk alloys, as well as some structure-properties relationships are discussed in the paper.

3. "An equivalent homogenized finite element model of TiAl/TiAlN multilayered coatings"

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Improving the properties of nanocrystalline metal nitride coatings remains a challenge in metalworking industries, to lengthen tool life and increase cutting performance. One solution consists in depositing metal/nitride multilayered coatings to take advantage of the properties of the nitride, the metal and the interfaces to improve the fracture toughness while maintaining very high hardness. Reactive Gas Pulsing Process (RGPP) is a sputtering technique which allows to easily modulate the stacking of a coating at the nanometer scale. Ti0.67Al0.33 and Ti0.54Al0.46N (labelled TiAl and TiAlN) monolithic coatings were deposited by radio-frequency magnetron sputtering from a single titanium/aluminium target. (TiAl/TiAl)n multilayered coatings, close to 2 µm-thickness, were deposited by RGPP with several n stacking. Identification of the material's elasto-platic behaviour of TiAl and TiAlN was performed using experimental indentation of monolithic coatings for the elastic part. The plastic behaviour was determined using Finite Element Model Updating (FEMU) method from dual indentation technique. A finite element model of (TiAl/TiAl)n nano-multilayered coatings reproducing the mechanical behaviour during nanoindentation tests was developed using an equivalent monolithic coatings with an elasto-plastic behaviour considered as a mixture law of both TiAl and TiAlN monolithic properties. So only one parameter, the equivalent volume fraction Vf of TiAlN, is necessary to define a n multilayered coating and to reproduce both the experimental P(h) curves and the hardness. The obtained Vf for each nano-multilayered coatings are compared with the experimental data obtained by Electron Energy-Loss Spectroscopy at the N-K-edge.

4. "Determining The Flexoelectric/Photo-Flexoelectric Response of Strontium Titanate Cantilevers by Nanoindentation Instrumentation"

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Flexoelectricity is a phenomenon where an electric polarization is induced in a material due to a non-uniform strain gradient. Unlike other phenomena like piezoelectricity and ferroelectricity, this effect is not limited by specific geometries or crystalline structures, which makes it significant in energy harvesting. Furthermore, it is possible to generate the so-called photo-flexoelectric effect in perovskite materials, both inorganic and organic, as the flexoelectric effect can be enhanced by the photocurrent generated in these materials. Flexoelectricity is currently characterized by methods such as Dynamic Mechanical Analyzer or techniques based on Scanning Probe Microscopy. The main drawbacks of those methods are the limited achievable oscillation amplitude of a contact tip and the relatively large size of the sample. In this work, we will show a novel, unique method using the Nanoindentation Instrumentation and free-standing cantilever beams. Using

direct mechanical excitation, we will experimentally show the flexoelectric response of a single strontium titanate crystal (STO) obtained by sub-micrometre oscillations. Furthermore, will utilize LED light to investigate the photo-flexoelectric effect induced by the monochromatic light irradiation of different wavelengths. Contrary to the commonly used methods, the presented method allows access to a relatively small strain field and proves effective for investigating the responses of significantly smaller materials.

[1] E. Coy, Method for probing flexoelectric response of free-standing cantilever beams by nanometric oscillations with nanoindentation technique, Measurement (Lond) 163 (2020). https://doi.org/10.1016/j.measurement.2020.107986.

5. Characterization of surface and mechanical properties of microbial cells

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In recent years, interest in the mechanical properties of prokaryotic organisms has expanded. For this purpose, Cupriavidus necator and Rhodospirillum rubrum have been selected; strains of bacteria that possess the ability to accumulate storage substances called polyhydroxyalkanoates or PHAs in their internal compartment [1]. The goal is to understand these bacteria as much as possible in order to increase production and lower production costs for PHAs as biocompatible and biodegradable polymers. Mentioned cells were immobilized using poly-L-lysine on a glass substrate and subjected to analysis by atomic force microscopy (AFM) and nanoindentation in a liquid medium under physiological conditions. AFM measurements were carried out using JPK NanoWizard 4. MLCT – A or MLCT-SPH-1UM-DC – A tips with the spring constant around 0.1 N·m-1 were applied for measurements in liquid using QI™ mode. In this way, topographic maps of these cells were obtained, and the elastic modulus was also determined from the reached force-distance curves using Hertzian contact analysis. Nanoindentation measurements were performed using Hysitron BioSoft instrument in displacement control mode. Flat punch tips (Rc 20 and 50 μm) at 10 μm·s-1 were used for single-cell compression tests. Data were evaluated according to Overbeck model [2]. It was observed, that bacteria accumulating PHAs exhibit difference in height from bacteria non-accumulating PHAs. Results from AFM show the elastic modulus of each measured bacterial strain is significantly different, where obtained elastic modulus averaged up to 1 MPa, while the compression tests from nanoindentation provided higher values (up to 12 MPa).

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[2] A. Overbeck, S. Günther, I. Kampen a A. Kwade. Compression Testing and Modeling of Spherical Cells – Comparison of Yeast and Algae 40(6), 1158-1164, (2017).

6. "A robust and fast method to estimate cubic elastic constants based on nanoindentation and Bayesian Inference"

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Estimating the elastic constants of materials constitutes a real challenge, particularly when dealing with multiphase materials. For single-phase materials, scientists have used techniques such as ultrasonic pulse echo (UPE) or a combination of X-rays and diffuse thermal scattering. For multiphase materials, these techniques, if used alone, can only give the macroscopic elastic constants of the materials and cannot distinguish the elastic constants of a specific phase. In the present work, a new protocol is presented using a coupling of nanoindentation experiments, Vlassak and Nix model, and Bayesian inference to statistically estimate the elastic constants of cubic materials. The originality of our method lies in writing the cubic stiffness tensor not in terms of the usual constants but in terms of the directional Young's modulus E[100],the directional Poisson's ratio ν[100] and the Zener coefficient A. Interestingly, E[100] then becomes a pre-factor of the stiffness tensor so that indentation modulus ratios depend on just two parameters, A and ν[100]. Consequently, we have developed a Bayesian inference code that considers indentation modulus ratios and varies only A and ν[100]. This approach has many advantages: computation time is greatly reduced, the use of indentation modulus ratios increases the number of data and reduces the influence of the calibration parameters, the limits of ν[100] are known and those of A can be easily conjectured unlike the ranges of Cij constants, which are much harder to estimate a priori.

7. "Deformation mechanism of cerium oxide nanocubes studied by in situ nanocompression in Environmental TEM"

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Cerium oxide nanoparticles are used in many industrial products, such as in solid oxide fuel cell electrodes, catalysis, or as superior abrasive particles in chemical mechanical planarization. However, few studies dealt with the determination of their mechanical properties or their deformation mechanism. Using a dedicated Hysitron PI 95 sample holder, deformation experiments of CeO_x monocrystalline nanocubes (20-130 nm in size) are performed in an environmental transmission electron microscope (ETEM). The ETEM mode is used to control the environment around the nanocubes. Indeed, by changing either the gaseous environment (high vacuum or air) or the electron dose rate conditions (high or low dose rate), different phases of CeO_x are investigated: fluorite (space group Fm-3m) when x ranges between 1.75 and 2, or bixbyite (space group Ia-3) when x is less than about 1.75. Dislocations can be indexed using dark field imaging before and after compression, or using contrasts in bright-field images. The slip system 1/2[110]{111} is determined as the main one in fluorite CeO_x with the [001] compression axis. In addition, partial dislocations and stacking faults were evidenced in the bixbyite structure using HRTEM observations. This result may be explained by the dissociation of perfect dislocations due to the presence of oxygen vacancies in the {111} planes. According to the evolution of defects and yield stress changes, the phases have an influence on the mechanical properties and deformation mechanism in CeO_x.

8. “Quantification of Irradiation Damage in Nuclear Power Plant Structures through Indentation Size Effect Analysis”

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Nuclear irradiation damage is a complex process characterised by increased dislocation density, micro-voids formation, and the generation of dislocation loops, etc. Nanoindentation emerges as an attractive technique for studying materials intended for nuclear structural applications due to its ability to vary test size to study local plasticity changes due to intrinsic/extrinsic length scales. In a study on pure iron, Hou & Jennett modified the slip distance semi-empirical model used to investigate pre-damaged samples, i.e. mechanical strain and ion implantation. Indentation on these samples was performed using Berkovich indenter. The results from this semi-empirical model exhibit a strong correlation with experimentally measured damage densities obtained using TEM, providing valuable insights into the length scale effects induced by mechanical strain and ion implantation, thereby facilitating the evaluation of the material’s damage state in small sample volumes.

9. “Formation of nanoscale intergranular cavities: in situ SEM and simulations”

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We present surface deformation measurements obtained by in situ SEM tensile tests. They are obtained by tracking gold nano-droplets formed by laser dewetting of a thin film of gold. The resolution enables imaging fine slip bands with a thickness of the order of 150-250 nm and Von Mises deformation up to 80%. The material studied is Eurofer 97 for nuclear application where slip localization might be important for initiation of ductile fracture (by fracture of carbides or other mechanisms). We also investigate, by atomistic simulations, the generic problem of the formation of intergranular cavities by the aggregation of vacancies, in relation to fracture [1]. The “Smart Darting” method was adapted to simulate delocalized intergranular vacancies within Markov chain Monte Carlo [2]. The segregation of single and di-vacancies was studied in about 10 simple tilt grain boundaries both in Al and in bcc Fe. The new method is efficient and the study of di-vacancies already enables selecting which grain boundaries are more prone to host clusters of vacancies.

[1] “Cohesive stress heterogeneities and the transition from intrinsic ductility to brittleness” D. Tanguy, Phys. Rev. B 96 174115 (2017)
[2] “Sampling vacancy configurations with large relaxations using Smart Darting” D. Tanguy, Phys. Rev. Materials accepted (2024) https://hal.science/hal-04245426v1

10. “Creating a digital twin and how it helps to speed up your coating development”

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A better understanding of your coating stacks and their behavior in the application is crucial for the optimization of complex engineering systems. In this context, a modeling approach, in conjunction with targeted laboratory and functional tests, is particularly attractive as it can accelerate the coating selection and achieve its goals within the desired application field. Indeed such methodology helps to understand the coating system, including the substrate and all coatings and interfaces. Simulations can find coating limitations and guide the refinement of the coating architecture within a defined framework. A model which will contain as much digital information as possible about your sample - we call it a digital twin. This work will showcase how the required data can be obtained by analyzing indentation and scratch measurements. SIO developed analytical models which dramatically speed up the simulation and optimization of complex contact conditions. With the help of these models you can first dimension the relevant experiments. Afterwards you can use the experimental data from the indentation experiments to evaluate the true Young’s modulus and yield strength of each coating. By analyzing scratch tests you can calculate critical values like the tensile stress.

In the second part of this talk we show how the digital twin can help you to find the best coating architecture for a new application by using the gathered digital data. Even if the contact conditions for the new application are not known exactly, you can use the digital simulations to narrow down the applicable samples from your portfolio. Comparing the simulations with the results of your standard or benchmark tests could also help you to optimize the coating properties and increase the application performance.

11. "Towards bulk and surface mechanical properties of a polyimide sample using AFM and instrumented nano-indentation"

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Quantifying the mechanical properties of polymers used in microelectronics is crucial for their integration. In particular, precise knowledge of their thermal behavior is essential to prevent delamination during the fabrication steps. These measurements of Young's modulus and hardness are generally carried out using instrumented nano-indentation (NI). However, this technique is difficult to implement for thin layers (thickness < 200 nm) because of its lack of precision in contact point determination and its large indentation depth which requires to take into account the rigid substrate beneath the layer of interest. By contrast, performing nano-indentation with an Atomic Force Microscope (ni-AFM) allows probing the mechanical properties of thin films. In this work, we evaluate quantitative and correlative analyzes between ni-AFM and conventional NI in order to compare the top surface and bulk properties of a common sample. As a polymer of interest for both techniques, we chose a 6-µm thick polyimide layer on silicon with a very low surface roughness for compatibility with ni-AFM. Unlike conventional modeling of ni-AM based on Hertz [1] elastic theory, we used the Oliver & Pharr [2] elastoplastic approach. The mechanical properties obtained with NI and ni-AFM are in good agreement for the same indentation depths, paving the way for the routine use of this approach and allowing to link the surface and bulk properties obtained with these two measurement techniques.

[1] I.N. Sneddon, International Journal of Engineering Science 3, 47-57 (1965).
[2] W. C. Oliver and G. M. Pharr, Journal of materials research 7, 1564-1583 (1992).

12. "Investigation of the elastic properties of single Au nanoparticles by the coupling of insitu TEM nanocompression experiments and Brillouin spectroscopy"

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The measurement of acoustic vibrational modes is fundamental to the full comprehension of the mechanical properties of nano-objects, since they are connected to the intrinsic characteristics of the material, such as crystallinity, size, shape and elasticity. At the nanoscale, when localized plasmons couple to localized vibrations, the enhancement of inelastic light scattering signals from acoustic vibrational modes is expected. In this contribution, we investigate the acoustic vibrations of individual 80 nm Au nanocubes using low-frequency Raman spectroscopy. We show that changes in incident polarization affect the phonons and thus modify the distribution of the electrical field inside the particles, therefore modifying the efficiency of the scattering by the acoustic vibrational modes. We also perform in situ nanocompression experiments in the TEM on isolated Au nanocubes as a way to assess their mechanical properties. Comparison of results obtained from Brillouin spectroscopy and in situ nanocompression experiments will lead to a better determination of the mechanical properties of nanoparticles. Furthermore, a coupling of the PI 95 sample holder on the Brillouin spectrometer has be designed and is presented.

13. "Deformation mechanisms of inorganic fullerenes used as lubrication additives : an in situ TEM nanocompression study"

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Inorganic fullerene-like – IF- nanoparticles made up of metal disulphides MoS_2, WS_2 are considered as promising candidates for anti-wear and anti-friction additives with significant application potential[1]. Their functional mechanism relies on the exfoliation of the external layers of the nanoparticles under mechanical stress leading to the formation of low friction tribofilms[2]. Optimizing the tribological properties can be achieved by controlling the size, shape and chemistry of the IF nanoparticles In this study, MoS_2 nanoparticles with controlled sizes were prepared by exposing the sheelites $MoMO_4$ nano particles to a H_2S-CCl_4-Ar gas mixture[3]. The IF-MoS_2 nanoparticles dispersed and sonicated were deposited on silicon substrate mounted on a copper pallet attached to the Bruker/ Hysitron PI-95 TEM holder. In situ compression and friction behaviour of MoS_2 nanoparticles with different level of crystallinity (poor, medium, high) under vacuum and oxygen atmosphere were performed , in an aberration corrected environmentally controlled FEI-ETEM instrument. The structural changes of nanoparticles under compression and/or frictional forces were imaged in TEM-bright field mode. Significant events shown by the force-displacement curve were identified and the corresponding structural changes in the IF nanoparticle were analysed in detail. The evolving deformation mechanism and its significance on the performance of the IF will be discussed.

1 Spikes H. Tribology Letters, 60, 5 2015.
2 Cizaire, L., et al., Surface and Coatings Technology, 160, 282, 2002.
3 Afanasiev, P Appl. Catal. B: Environmental, 227, 44 2018.

14. "Static friction at the microscale investigated at the limit of Hertzian pressure by oscillatory shear experiments"

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The force to initiate relative motion between contacting bodies -the static friction force- has been investigated with a novel 2-dimensional nano-indenter setup (©KLA Gemini) for a single micrometer scale asperity. Both normal load and stiffness (contact area) are monitored while simultaneously applying an oscillatory shear force of controlled amplitude. A diamond spherocone tip (R= 5um, ©Synton) tested on a flat fused silica sample is shown, plotted as normalized lateral oscillatory load vs. lateral oscillatory displacement at increasing normal loads. A decrease from a full stick zone elastic response to fully sliding expected from Mindlin's sheared contact model can be seen as the falling slope of the curves. Accordingly, a static friction force is calculated by asymptote to zero stiffness. Static friction coefficients have been shown to be decreased by increasing load which we explain by correlating the friction force with interfacial shear strength (ISS) and contact area. Therefore, up to the onset of plasticity, the contact area and load obeys Hertzian relations that should yield a decreasing static friction coefficient by an inverse root cube proportionality to load. Furthermore, ISS and its dependency to normal load has been investigated by several molecular dynamics approaches that demonstrates a weak dependency of ISS to applied load.

15. “Nanoindentation of a rough surface“

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Instrumented indentation is a useful tool for measuring the properties of a sample’s superficial layer, from machined surfaces to thin coatings. However, the low thickness of this layer can make polishing impossible. Therefore, the question of rough surface indentation arises, as usual models [1] are developed for flat surfaces. Results are greatly scattered by roughness leading to the idea of a statistical analysis. This includes determining the necessary number of indents and whether it depends on roughness parameters [2]. The control of surface roughness by means of FemtoLASER shots allows for the study of mean hardness evolution with the number of indents for different roughness parameters, ultimately leading to the establishment of guidelines for rough surface indentation. For a deterministic approach, we use the Hysitron-TI-980 SPM mode to measure the sample surface before and after indentation. The evolution of contact area against roughness parameters can thus be modelized.

[1] Oliver, W.C., Pharr, G.M., J. Mater. Res. 7: 1564–1583, 1992
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16. “Key parameters affecting the local material characterization of interfaces in polymer blends: dialogue between AFM measurements and FEA simulations”

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With the increasing emphasis on recycling and the challenges associated with sorting polymers in particular, non-miscible polymer blends are arising as potential end materials. The associated scientific challenge is thus to better understand and predict the behavior of such materials. More specifically, in immiscible blends the role and properties of interfacial regions (interfaces with or without interphases) are crucial and need to be reliably characterized. To that end, the dialogue between AFM measurements and FEA linear elastic calculations is further pursued. Indeed, while AFM is a powerful tool essential for the microstructural and micromechanical characterizations of nanostructured systems, it remains a very much indirect measurement affected by multiple factors inherent to nanoindentation. Thus, through the association of experimental AFM measurements and FEA simulations, we will attempt to understand the key metrics triggering the modulus reduced from AFM measurements, and how, for instance, it is affected by varying parameters such as geometrical ones (the surface topology, the interface orientation …) and local material properties (phases and interphases). Now focusing on polymer systems, attention will also be devoted to a more refined description of their small strain mechanical behavior, namely the account for the time dependent viscoelastic response.

17. “How to perform micro-bending beam push and pull fatigue during electrochemical hydrogen charging in scratch test mode”

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As climate change progresses, the need for decarbonization poses new challenges for materials. Exposure to hydrogen in structural materials and components is expected to increase in the coming years. It is well known that the interaction of hydrogen with materials is complex and depends significantly on the material itself and its state. Therefore, the importance of highly localized test methods for the hydrogen sensitivity of materials, especially in multiphase systems, is evident. Over the past two decades, electrochemical nanoindentation (EC-NI) has provided deep insights into material behavior. The investigation of fatigue crack

growth in microbending beams was previously limited to applications in the scanning electron microscope (SEM), if negative stress ratios (push and pull) were to be applied. However, this prevented simultaneous hydrogen charging with high hydrogen concentrations by electrochemical high fugacity charging. We present a novel implemented method that allows to fatigue a microbending beam fabricated by focused ion beam (FIB) in a liquid and thus in an EC cell using a gripper. We use a WC tip, which we attach to the 2D transducer of a TI980 with an electrically insulating Macor holder. The gripper is cut onto this tip using FIB. The gripper is then positioned over the beam in the EC cell via SPM scanning, lowered and fatigued using "air scratch".

18. "Measurement of resistance in solid laden liquids – Initial traverses at room temperature"

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Force-displacement interactions in liquids and semi-solids are non-conventional for nanoindentation given the preconditions of finding a surface before testing. The nanoindentation system provides extensive freedom in conducting tests with just a few mandatory requirements. For undertaking tests, different readily available solid entrained liquids - Philadelphia cream cheese, butter and Nutella – were taken as tests samples to represent water entrained solid and oil entrained solid masses. Initial tests were undertaken directly on the solid laden liquids at room temperature to observe the tip to solid surface interaction. A new approach was developed allowing initial recognition of a surface before engaging in the test. Traverses at a shallow depth to represent the top surface and deeper into the sample were undertaken to discern differences in behaviour. Just like initial contact with the sample is important, so removal of the tip is important at the completion of testing. Initial results will show the interaction and resistance to spreading for different solid laden liquids, and the interaction at the completion of the experiment.

Practical Information

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Nanobrücken 2024 will be held at the Hôtel Valpré-Lyon, located in the beautiful city of Lyon.

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For additional information, please visit the conference website at 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

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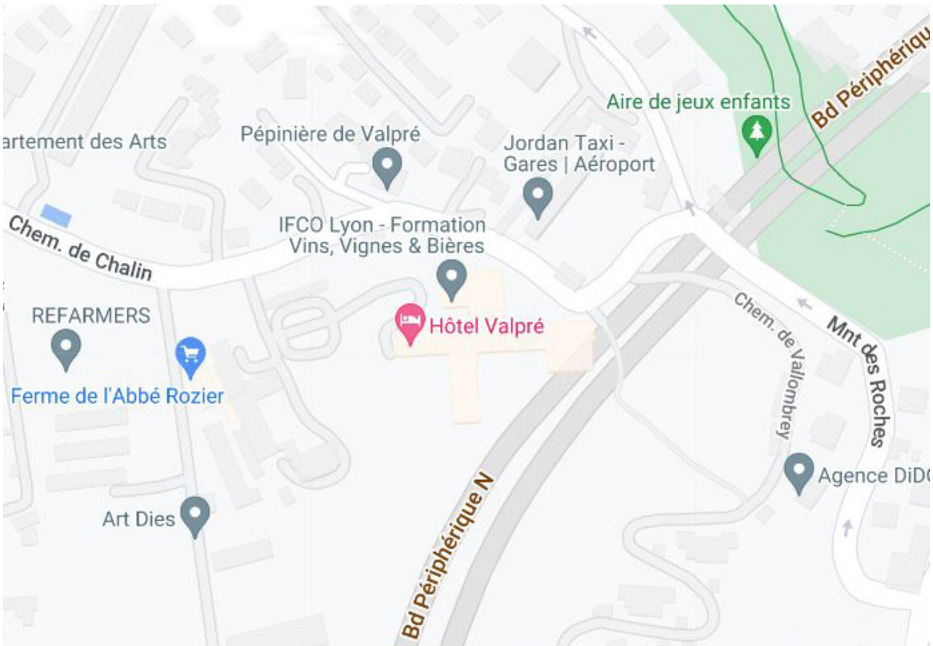

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