Nanobrückchen 2021

Nanomechanical Testing Conference

February 23–24, 2021 | 16:00 - 21:00 CET / 9:00AM - 2:00PM CST

Virtual Event, Actual Science
Preface

Bruker is pleased to announce that Nanobrück 2021 is going virtual on February 23–24, 2021. This free on-line conference will provide attendees the opportunity to engage with high-quality technical programming and industry leaders in a safe and healthy environment.

Nanobrück, Bruker’s annual conference for international researchers and industrial leaders in nanomechanical and nanotribological testing, includes oral presentations from leading research groups, as well as live demonstrations and discussions with Bruker experts. The nanomechanical testing conference also features a lively competition for best student video contribution and best poster.

Topics will include advanced nanoindentation and associated techniques, testing in extreme environments, in-situ testing in SEM/TEM, theory/simulation and biomechanical testing.

Practical Information

Conference Webpage
https://www.bruker.com/nanobruecken

Sponsor Website
Bruker: https://www.bruker.com

Presentation Times
The presentation times are listed in Central European Time (CET) and Central Standard Time (CST).
Day 1: Tuesday, February 23

16:00 | 9:00 am Welcome and Introduction
Douglas Stauffer (Bruker)

16:05 | 9:05 am Neural Networks Capture the Deformation of Lattice Metamaterials
Brad L. Boyce (Sandia National Laboratories)

16:30 | 9:30 am Revealing the Lattice Scale in Microcompression of Nb
Robert Maaß (Federal Institute of Materials Research and Testing (BAM) and University of Illinois at Urbana-Champaign)

17:00 | 10:00 am Hysitron TI 980 Demonstration with Eden Prairie and Aachen Labs
Ude Hangen, Radhika Laxminarayana, and Jungkyu Lee (Bruker)

17:30 | 10:30 am Poster Session

18:00 | 11:00 am Indentation Strain Rate Effects in Advanced Materials and Alloys
Nathan A. Mara (University of Minnesota)

18:30 | 11:30 am Combined Assessment of Bone Material Quality Using a Novel, Integrated Nanoindenter-Raman Spectroscopy System
Virginia L. Ferguson (University of Colorado at Boulder)

19:00 | 12:00 pm Live from the Eden Prairie Lab
Lance Kuhn, Justin Patten, and Douglas D. Stauffer (Bruker)

19:30 | 12:30 pm Sharing Solutions with our Product Marketing Manager David Vodnick: A Look into the Nanomechanical Test Systems
David Vodnick (Bruker)

20:00 | 1:00 pm Meet & Greet with General Manager Oden Warren and Bruker’s Hysitron Team

20:00 | 1:00 pm Poster Session

21:00 | 2:00 pm End of Session

Day 2: Wednesday, February 24

16:00 | 9:00 am In-Situ Nanoindentation of Air Sensitive Materials for Battery Applications
Ed Darnbrough (University of Oxford)

16:30 | 9:30 am Ultrahigh Temperature Small Scale in situ TEM based Mechanical Testing
Shen Dillion (University of Illinois Urbana-Champaign)

17:00 | 10:00 am Hysitron PI 89 Demonstration with Eden Prairie Lab
Eric Hintsala and Praveena Manimunda (Bruker)

17:30 | 10:30 am Tech Talks with Bruker
Meiken Falke and Thomas Mueller (Bruker)

18:00 | 11:00 am Towards Mechanical Characterization of Nano-Scale Interfaces in Biological Materials
Igor Zlotnikov (B CUBE – Center for Molecular Bioengineering)

18:30 | 11:30 am Mixed Mode Cracking of Martensitic Sharp Edges: An In-Situ SEM ‘Shaving’ Study
C. Çem Tasan (Massachusetts Institute of Technology)

19:00 | 12:00 pm Sharing Solutions with Product Marketing Manager Sanjit Bhowmick: A Look into the Nanomechanical Instruments for SEM/TEM
Sanjit Bhowmick (Bruker)

19:30 | 12:30 pm Awards and Announcements

20:30 | 1:30 pm End of Session
Video Poster List

Ordered by last name of presenting author

1. Micromechanical spectroscopy as a tool to measure defect relaxation in nanostructured metals
   Markus Alfreider (Montanuniversität Leoben)

2. On the mechanical properties and thermal stability of Zr$_x$Cu$_{100-x}$ thin film metallic glasses with different compositions
   Andrea Brognara (Max-Planck-Institut für Eisenforschung GmbH)

3. An innovative in-situ multi-tool rotational 4-point-bend system for micro-mechanical stress impact analysis
   André Clausner (Fraunhofer IKTS)

4. High ductility of amorphous olivine at room temperature during in situ nanomechanical tensile testing in the TEM
   Patrick Cordier (University of Lille)

5. Contact area correction for surface tilt in pyramidal nanoindentation
   Joseph E. Jakes (USDA Forest Service, Forest Products Laboratory)

6. pyNIDA – open-source software for the graphical data analysis of in-situ indentation
   V.A. Lebedev (University of Limerick)

7. Microscale deformation and fracture studies in Barium Titanate
   Nidhin George Mathews (Indian Institute of Technology Bombay)

8. Direct determination of the area function for nanoindentation experiments
   Christian Saringer (Montanuniversität Leoben)

   Jendrik Silomon (Volkswagen AG)

10. In situ observation of dislocation evolution in cerium oxide nanocubes in an environmental TEM
    Rongrong Zhang (INSA-Lyon)
PDF Poster List
Ordered by last name of presenting author

1. Strength of interface in bioresorbable poly-lactic acid/Mg fiber composites for orthopedic applications
   Wahaaj Ali (IMDEA Materials)

2. Screening of solvents for 2D materials exfoliation using nanoindentation
   Zainab Alkharusi (University of Manchester)

3. Acquisition of Residual Stress and Fracture Toughness of Metals Through Nanoindentation
   Ömer Necati Cora (Karadeniz Technical University)

4. Single Crystal Thin Film Mechanical Behavior Measured with Nanoindentation
   M.J. Cordill (Erich Schmid Institute for Materials Science)

5. In-situ electrochemical nanoindentation as a promising tool for probing hydrogen-material interaction
   Anna Sophie Ebner (Montanuniversität Leoben)

6. Nanoindentation study of dislocation-based plasticity and crack formation in oxides
   Xufei Fang (Technical University of Darmstadt)

7. The Effect of Loading Strain Rate on Nanoindentation Response of RTM6 Epoxy Polymer: Experimental and Computation Research
   Patricia M. Frontini (University of Mar del Plata)

8. In-situ TEM investigation of toughening in Silicon at small scales
   Inas Issa (Montanuniversität Leoben)

9. Fracture behavior of a tough bulk metallic glass at micrometer dimensions
   A. Jelinek (Montanuniversität Leoben)

10. Probing structure-property relationships in Cu-Ni and Cu-Zn alloys in nanofoam form with nanoindentation
   Alexandra Loaiza (Purdue University)

11. Mechanical property determination for cutting tool and workpiece using indentation and investigations on the boundary film formed in sliding contacts with emulsions
    Florian Pape (Leibniz Universität Hannover, IMKT)

12. Micro-scale indentation on active microelectronic circuits to determine the piezoresistive coefficients of transistor channels
    S. Schlipf (Fraunhofer IKTS)

13. Automated crack advancement detection for small scale fracture experiments
    Klemens Schmuck (Montanuniversität Leoben)

14. Using machine learning algorithms to interpret finite element simulations of indentation experiments including tip radii effects
    Claus Trost (Erich Schmid Institute of Materials Science)

15. Fatigue Tests of Penta-twinned Ag Nanowires under TEM and Their Structure Analysis
    Hu Zhao (University of Manchester)
Abstract - Oral Presentations

Day 1: Tuesday, February 23

16:05 CET | 9:05am CST

Neural Networks Capture the Deformation of Lattice Metamaterials

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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.


16:30 CET | 9:30am CST

Revealing the Lattice Scale in Microcompression of Nb

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Intermittent microplasticity via dislocation avalanches indicates scale-invariance, which is a paradigm shift away from traditional bulk deformation. Recently, we have developed an experimental method to trace the spatiotemporal dynamics of correlated dislocation activity (dislocation avalanches) in microcrystals (Acta Materialia 122 (2017) 109; Acta Materialia 152 (2018) 86). Here we apply this approach to temperature-dependent micro-compression of Nb single crystals. We focus on the changes of the discrete plastic flow response between 370 and 170 K. Whilst the flow stress as a function of temperature is in excellent agreement with thermally-activated bulk plasticity models for bcc metals, a temperature insensitive component is revealed when tracing the change of displacement discontinuities with decreasing temperature. It is found that the scale of the displacement jumps changes from a scale-free like behavior at high temperatures to a scale-dependent behavior at the lowest temperature, where the length scale converges towards the scale of the lattice parameter. 3D discrete dislocation dynamics simulations are done to shed light onto the underlying dislocation behavior, revealing that the athermal plasticity component is governed by screw-dislocation activity. The temperature insensitivity of this screw-dominated plasticity can be shown to depend on high local stresses, that significantly exceed the Peierls stress in the studied Nb microcrystals. This work is the most recent cornerstone of our efforts to understand materials-dependent and non-trivial stress-strain fluctuations in microplasticity (Phys. Rev. Mat. 2 (2018) 120601; Phys. Rev. Mat. 3 (2019) 080601).
18:00 CET | 11:00am CST

Indentation Strain Rate Effects in Advanced Materials and Alloys

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In recent years, the ability of instrumented indentation to carry out indentation strain rate jump testing (SRJT) has opened new opportunities to probe the strain rate dependency of material deformation. Such testing allows for the determination of hardness as a function of indentation depth and strain rate via CMX test protocols, allowing for calculation of the deformation mechanism-dependent key parameters of strain rate exponent, activation volume, and activation energy. Here, I will present our recent work utilizing SRJT protocols to investigate the deformation rate dependency of three materials: pure W, advanced nuclear reactor steels, and microcrystalline cellulose. We find that utilizing Bruker’s modified TriboIndenter 980 enclosed in a vacuum chamber with a custom load function generator that we can conduct jump testing between rates of ~5x10^-4 s^-1 and ~0.5 s^-1, and temperatures up to 800°C to determine the rate dependent parameters for creep of T91 ferritic-martensitic steel alloy.

18:30 CET | 11:30am CST

Combined Assessment of Bone Material Quality Using a Novel, Integrated Nanoindenter-Raman Spectroscopy System

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Biological materials possess an exquisite heterogeneity that spans multiple hierarchical levels and across length scales. Material heterogeneity takes on a range of forms that includes variability in the organization tissue's underlying extracellular matrix and biochemical composition. Bone is one of the most commonly indented tissues, yet the contribution of material heterogeneity is often overlooked in its measurement. In contrast, the bone research community has recently embraced the need for material heterogeneity as a toughening mechanism that inhibits fracture propagation. Clinical manifestations exist where impaired bone “tissue material quality”, with heterogeneity that is low or too high, leads to increased fracture prevalence when bone material becomes homogeneous or too variable. Nanoindentation is an ideal tool to evaluate the variability of indentation modulus in bone and other tissues. Yet nanoindentation alone provides information that fails to provide context as to the source of heterogeneity. Here, we detail how analysis of bone heterogeneity and general materials characterization is improved through the integration of a Renishaw InVia confocal Raman spectroscopy system. Fiberoptic probes enable assessment of materials at indent sites using the optics of a Hysitron TI 950 Triboindenter. Standardization of this process was performed by evaluating pressure induced phase transformations in Si, and then used to evaluate bone tissues from two studies of bone. In the first, we established a direct correlation between tissue chemistry and indentation modulus in bones from mice flown in microgravity for ~14 days. We next used this system to explain how bone heterogeneity influenced fracture toughness in a study of aging, high fat diet, and exercise in rats. Notably, combined assessment revealed that the heterogeneity of bone tissue modulus could explain fracture toughness, but only when taking into account the chemistry of the bone tissue. This novel result adds to our understanding of how bone material quality relates to propensity for fracture.
Abstract - Oral Presentations
Day 2: Wednesday, February 24

16:00 CET | 9:00am CST

In-Situ Nanoindentation of Air Sensitive Materials for Battery Applications

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New battery technology is key in order to democratize access to Energy across the world and to help the developing nations leap-frog a carbon-based industrial revolution to save our futures. The challenges where most progress is being made is in understanding failure mechanisms and speculation on the role of mechanical properties. The lack of reported mechanical properties for battery materials is due to; their reactivity with oxygen/water/nitrogen (needing specialised equipment), the small volumes of material available for testing and the difficulty in producing homogeneous fully dense samples. This makes in-situ nanoindentation an exciting opportunity to make an impact in an important research area.

In this talk I will introduce the work being done at Oxford to overcome these material challenges to generate mechanical property data that are useful for modeling in cycling effects.

16:30 CET | 9:30am CST

Ultrahigh Temperature Small Scale in situ TEM based Mechanical Testing

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Small-scale mechanical testing has greatly impacted understanding of materials mechanics across broad classes of materials chemistries and systems. Much of the work, however, has been performed in a relatively narrow temperature window close to ambient temperature. The mechanical properties and deformation mechanisms of refractory materials are often of primary interest at high homologous temperatures. Combining small scale mechanical testing with localized heating sources, such as laser heating, enables mechanical testing into the ultrahigh temperature regime. The approach, in fact, can access temperature regimes where conventional bulk mechanical testing is not feasible. The methodology can, furthermore, exploit mechanical coupling, simple sample geometries, and temperatures where diffusion is facile to characterize thermodynamics and kinetics of diffusive processes that are challenging to measure using conventional materials characterization techniques.

This talk will discuss some of the challenges and opportunities associated with small scale mechanical testing performed at high temperatures resulting from localized laser heating in the transmission electron microscope (TEM) using a Hysitron PI 95. Practical aspects of the experiments, such as laser alignment, temperature calibration, environmental constraints, and sample constraints will be discussed. Examples provided include characterization of high temperature ion irradiation induced creep in single crystalline and nanograinced alloys, and nanopillar compression and bicrystal tensile Coble creep in oxide systems. In the latter examples, it is demonstrated how the approach can be used to measure grain boundary, interphase boundary, and surface diffusivity, along with surface energy, grain boundary point defect formation volumes, and grain boundary activation volumes.
Towards Mechanical Characterization of Nano-Scale Interfaces in Biological Materials

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Identifying the property-structure-function relationships in mineral-organic biocomposite materials is one of major challenges in today’s biomaterials science that incorporates research efforts in biology, chemistry, physics and engineering. The cross-disciplinary interest in the topic stems from the efficiency of the biochemical machinery that is responsible for biotic mineral formation, the unconventional functional capacity of these tissues and, at the same time, elegance and even simplicity of “engineering” solutions it provides to the organisms. Specifically, nature is successful in forming complex hierarchical biocomposites with superior mechanical properties that provide the animals with high stiffness, high toughness and in some cases, are adapted for functional requirements that involve viscous damping, such as impact absorption, signal filtering and vibrations inhibition. In highly mineralized tissues, the stiff and hard mineral building blocks at all hierarchical levels are usually joined together by ultra-thin compliant, soft and viscoelastic organic interfaces that, in some cases, are only few nanometers thick. Although these interfaces comprise merely a small volume fraction of the biocomposite structures, the performance of the entire tissue is considered to be substantially affected by their mechanical characteristics. However, our understanding of the contribution of the organic interfaces to the mechanical functionality of these biocomposite assemblies is limited, mainly, because we still lack the capacity to assess their nano-mechanical properties.

The presentation will provide an overview of our recent achievements in developing experimental-numerical and experimental-analytical strategies to characterize the viscoelastic properties of organic interfaces in biocomposite architectures.

Mixed Mode Cracking of Martensitic Sharp Edges: An In-Situ SEM ‘Shaving’ Study

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Abstract will be available in final program.
Abstract - Video Poster Presentations

1. Micromechanical spectroscopy as a tool to measure defect relaxation in nanostructured metals

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Nearly all commonly available materials are of crystalline nature with respective inherent defects, e.g. point defects, dislocations, grain boundaries (GBs), which influence their physical properties. These defects can lead to desired or unwelcome changes in mechanical, electrical or thermal response. For example, severe plastic deformation decreases the grain size, while introducing dislocations and vacancies, leading to materials with a high content of all defect types exhibiting sometimes uncommon behaviour, e.g. hardening by annealing. Such characteristics are ascribed to local changes in GB structure. However, quantification of these phenomena rooted in the atomistic structure by atomic resolution imaging remains challenging. Therefore, it is necessary to search for alternative methods to investigate such problems.

In the present work, ultra-fine grained tantalum, processed by high pressure torsion and annealed up to 400°C, was studied by macroscopic hardness testing, micropillar compression and a novel method utilizing mechanical spectroscopy on micron sized specimens (µMS). The µMS-technique is based on the oscillating system of a Hysitron PI 85 with a nanoDMA III upgrade, which has a resonance frequency of ~114Hz. Changes in resonance frequency as well as damping capability in contact with the microcantilever shaped specimens can be studied and correlated with established techniques. For the investigated tantalum, an increase in hardness as well as yield onset and flow level during micropillar compression was observed upon annealing, while µMS showed a decrease in damping capability. Considering established models for dislocation damping as well as GB structure, the µMS data leads to the conclusion that local changes in GBs are the origin for this ‘hardening-by-annealing’ phenomenon.
2. On the mechanical properties and thermal stability of \( \text{Zr}_x \text{Cu}_{100-x} \) thin film metallic glasses with different compositions

**Andrea Brognara**¹,*, James P. Best¹, Philippe Djemia², Damien Faurie², Matteo Ghidelli¹², Gerhard Dehm¹

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Bulk Metallic glasses (BMGs) are metallic alloys characterized by a liquid-like atomic structure enabling a combination of superior mechanical properties - i.e. yield strength (~2 GPa) and high elastic deformability (~2%) - with large corrosion resistance and biocompatibility [1, 2]. However, BMGs suffer of a macroscopically brittle behavior induced by the formation of shear banding phenomena. Recent studies have shown how this behavior can be mitigated by reducing their intrinsic size through developing thin film metallic glasses (TFMGs, thickness < 1 μm), which report the suppression of shear banding process together with the mutual combination of large plastic deformation (> 10%) and yield strength (~3500 MPa, close to the theoretical limit) [3,4]. Nevertheless, several scientific challenges dealing with their mechanical properties and thermal stability are still open, especially focusing on the role of the composition addressing key questions involving the effect of local order, bond strength and free volume.

In this context, we investigated a large variety of binary \( \text{Zr}_x \text{Cu}_{100-x} \) TFMG compositions (24 < at.% <61), while investigating their mechanical and thermal behavior. Films have been deposited by magnetron sputtering, achieving a fine control of the composition by accurately tuning the applied power. X-Ray diffraction (XRD) has been employed to analyze the amorphous structure as well as its behavior as a function of temperature (with in-situ XRD heating). We find different crystallization temperatures, with the maximum value of ~380°C for \( \text{Zr}_{52} \text{Cu}_{48} \) composition which maximizes the number of mixed Zr-Cu bonds and the mixing enthalpy (\( \Delta H_{\text{mix}} \)), promoting the stability of the amorphous phase.

The elastic constants have been extracted by Brillouin light scattering and we show that all elastic moduli (Young’s, shear and bulk) increased with content of Cu (at. %), while Poisson ratio remained constant around 0.38. A similar trend is found for the hardness measured by nanoindentation, reporting an increment from 5.5 up to 7.7 GPa for Cu-rich specimens showing a closer atomic distances and larger bond strength. Nevertheless, the loading rate dependency analysis revealed that larger pop-in’s appeared at low Cu (at.%) content (for indentation rates below 25 mN/s), connected to a more disordered atomic structure with less strong atomic bonds enabling the formation of annular shear bands around the indent. Finally, films deposited on flexible polymeric substrates were used to study crack evolution during tensile deformation tests. Both crack and buckling density displayed relevant differences as a function of composition, with increasing resistance towards crack nucleation for samples with larger Cu at.% content, involving the highest bond strength. Highest maximum elongation of 2% and 13% respectively before cracks and buckle appearence, were reached for \( \text{Zr}_{24} \text{Cu}_{76} \).

Overall, the presented results highlight the effect of film composition on mechanical properties and thermal stability for \( \text{Zr}_x \text{Cu}_{100-x} \) TFMGs with potential interest for a variety of applications such as MEMS (Micro Electro Mechanical Systems), biomedical tools or flexible electronics.

**References:**

3. An innovative in-situ multi-tool rotational 4-point-bend system for micro-mechanical stress impact analysis

André Clausner¹*, Christoph Sander¹, Frank Macher¹, Simon Schlipf¹, Wieland Hein¹, Martin Gall¹, Ehrenfried Zschech¹

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A novel innovative in-situ multi-tool 4-point bending system using a patented rotational sample gripper geometry was developed at Fraunhofer IKTS. Its versatile design enables to characterize the influence of mechanical surface stresses on for example microelectronic devices, thin film systems, novel materials, and many more. Due to the low build height, sample manipulation design, and vacuum compatibility of the system, it can be used in-situ in several tools like nanoindentation systems, SEMs, or Raman microscopes. Because of its capability to preserve the stress state without power supply, correlative studies using different in-situ experimental approaches are enabled. Due to the homogeneous bending moment between the inner support points, the sample region that can be studied under stress is relatively large. Additionally, based on the innovative sample mounting, tensile and compressive stress can be alternatingly applied on the specimen surface without changing the setup and unmounting the sample enabling e.g. Low-Cycle Fatigue studies. Using this system, several application studies have been realized. For example, for ULK dielectric thin films on silicon, used in microelectronics, the influence of the surface stress state on the corner cracking behavior of cube corner indents have been studied using in-situ 4PB stressed experiments in both, a Hysitron TI 950 system as well as a SEM. The results from these experiments were used to parameterize a Cohesive Zone Model (CZM) in FEM simulations of the cohesive failures of these materials. Other experiments have been done in Raman microscopes as well as studying the influence of microscopic stress states on active transistor structures.

4. High ductility of amorphous olivine at room temperature during in situ nanomechanical tensile testing in the TEM

Patrick Cordier¹²*, Andrey Orekhov³⁴, Ralf Dohmen⁵, Michaël Coulombier³, Thomas Pardoen³, Dominique Schryvers⁴, Hosni Idriss³⁴

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(Mg, Fe)₂SiO₄ olivine is the main constituent of the upper mantle of the Earth. A recent study has shown that under low-temperature, high-stresses conditions olivine deforms by grain boundary sliding (GBS) along amorphized grain boundaries. The rheology of amorphous olivine (hereafter referred to as a-olivine) therefore appears to be determinant for grain boundary sliding under those conditions. However, amorphous olivine is very difficult to obtain from quenching the melt and is not found in nature. It is only obtained following or compression in diamond anvil. For this study, we used another processing method, pulsed laser deposition (PLD), which allows the deposition of amorphous thin films of olivine composition. In-situ TEM uniaxial tensile experiments were performed at room temperature on these a-olivine thin films using the Hysitron PI 95 TEM Picolndenter from Bruker. Despite a temperature which is very low for an amorphous silicate, the specimens are ductile (up to 25% strain) under high tensile stresses (3 GPa). We show here that this ductility can be further enhanced (up to 80%) by exposing the specimens to the electron beam of the TEM. Our results highlights an immense application potential of nanomechanics for fabrication of nanodevices.
5. Contact area correction for surface tilt in pyramidal nanoindentation

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A correction is needed to minimize errors in contact area caused by surface tilt in nanoindentation. Surface tilt decreases the contact area calculated using standard analyses that assume the tested surface is not tilted and thus results in overestimation of hardness and elastic modulus. Both the direction of tilt with respect to the pyramid face and the angle of the pyramidal probe are important when characterizing error caused by surface tilt. Here, a geometric model was used to create contour plots from which the area correction factor can be directly determined using only the ratios of side lengths measured from an image of the triangular nanoindentation impression. Contour plots for Berkovich, cube corner, and two nonstandard pyramidal probe geometries are given. The efficacy of the method was demonstrated in the correction of Berkovich nanoindentation on facets in freshly prepared poly(methyl methacrylate) with surface tilts as high as 6°.

6. pyNIDA – open-source software for the graphical data analysis of in-situ indentation

V.A.Lebedev¹,* , A.S.Poluboiarinov², D.A.Kozlov², R.M.Sakaev², V.I.Chelpanov³, Ya.Yu.Philippov², A.A. Krasilin⁴, A.V. Ankudinov⁴, A.V. Khokhlov², A.V.Garshev²

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Nowadays, the modern equipment allows one to perform in-situ indentation tests during the simultaneous observations by SEM or (S)TEM. Meanwhile, there is not so much free software available for the processing of the corresponding graphical data. pyNIDA is the open-source (GPL-3) Python-based software, which allows one to perform the full cycle of in-situ nanoindentation data processing.

First part of the software allows one to perform digital tracking of the object displacements observed by microscope by Digital Image Correlation functions from OpenCV. It is also possible to take a displacement of the whole system into the account tracking the substrate. The result of this part is a csv file with an observed object displacement vs. time. Second part involves a comparison of indentor displacement data and the results of visual observations. It allows one to calculate a sample drift function with respect to the zero point differences and scale factors. This part results in a drift function and in a csv file with drift-corrected load and displacement data vs. time. Third part serves for the analysis of the load-displacement data by one of the proposed models: Hooke's law or Hertzian model for sphere/cylinder. Fourth part is designed for the analysis of the particle shape evolution in time. Two different approaches are in use: contours analysis by OpenCV, and random walker segmentation of areas by skimage. For the moment this part has been properly tested for spherical particles only, but it is still under development.

Code is freely available by the link https://github.com/LebedevV/pynida. Your feedback is very important for us, so feel free to send your comments, opinions, and patches.

All test data were collected on MEMS-based Hysitron PI 95 at Zeiss Libra 200MCTEM.

This work was partially supported by the Russian Science Foundation (Grant 19-13-00151).
7. Microscale deformation and fracture studies in Barium Titanate

Nidhin George Mathews1,*, Ashish Kumar Saxena2,4, Christoph Kirchlechner2,*, N Venkataramani1, Gerhard Dehm2, Nagamani Jaya Balila1

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Barium Titanate (BTO) is a widely accepted lead-free piezoelectric ceramic used at micron length scales as thin films in MEMS applications. The dependence of a material's mechanical behaviour on the length scale of its application, typically called size effects, motivates us to study the deformation behaviour of BTO in its miniature form. Here we study the mechanical behaviour BTO single crystals and thin film systems using different micromechanical experiments and finite element modelling (FEM). Microscale mechanical behaviour of single crystalline BTO was studied by uniaxial in situ micropillar compression. It was observed that pillars below 1μm diameter reached their theoretical strength whereas larger pillars yielded at lower stress values with multiple stress drops confirming slip activity. The strain accommodation mechanism at smaller length scales is by plastic flow, with a size exponent close to 1 and enhances the elastic strain limit of the material, which is an important consequence that can be exploited in sensors/actuators. Microcantilever fracture tests were carried out on both single crystal and thin film forms. Various geometric aspects of microcantilever bending were modelled to optimise beam dimensions. Microcantilever fracture measurements revealed that, single crystal BTO showed a 45% higher $K_{IC}$ than the bulk, while the polycrystalline thin film showed a 60% lower $K_{IC}$ due to the weak inter-columnar boundaries. The reduced damage tolerance of thin films have implications in its application and needs further study with varying process parameters to achieve oriented films.

8. Direct determination of the area function for nanoindentation experiments

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The determination of a suitable correction for tip blunting is crucial in order to obtain useful mechanical properties from nanoindentation experiments at low indentation depths. While typically the required area function is acquired from the indentation of a calibration material, the direct imaging by suitable methods is an interesting alternative. In this work we demonstrate the applicability of confocal laser scanning microscopy (CLSM), atomic force microscopy (AFM) and self-imaging by scanning a sharp silicon tip with the nanoindentation tip and compare the results to the area function obtained by the indentation of fused. The important tip characteristics including the area function, the face angles of the three faces, the equivalent cone angle and the tip radius at the apex were determined by various methods based on the analysis of the obtained 3D data sets. It was found that the suitability of CLSM and AFM depend on the resolution and the operation mode, respectively. While for these methods only limited consistency of the determined tip characteristics was found, self-imaging resulted in an excellent overall agreement. Furthermore, indentation experiments on fused silica showed that below 50 nm a correction with the area function obtained by self-imaging was able to reveal the hardness decrease resulting from tip rounding which was not reflected by the results when the area function was acquired by indentation. In addition to assessing the suitability of different direct tip imaging methods, the work thus demonstrates the potential superiority of direct imaging methods.
Development and Application of a Customized Approach Combining Micromechanical Testing and Acoustic Emission

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To ensure the structural integrity of any component in the field, thorough mechanical testing should be applied beforehand. To make precise predictions regarding damage occurrence and propagation processes it is necessary to identify critical loading parameters and understand the related damage modes. In this work, a customized approach is presented to evaluate mechanical damage modes occurring in a heterogeneous material stack of a semiconductor sample. For applications in this field standards [1] and experimental approaches [2] are available to test the mechanical reliability of the stack by applying mechanical load to the adjacent electrical connectors, e.g. solder balls or Copper pillars (Cu-pillars). These approaches are based mainly on the evaluation of mechanical parameters. In this work, the additional usage of acoustic emission (AE) signals is suggested. AE measurements can serve two purposes: It can be utilized for damage indication to determine if and when a damage occurs, but also to obtain further information regarding the damage event. This is complementary to the mechanical measurement results. AE data can for example be utilized to categorize occurring damages [3,4].

The experiments presented in this work were conducted utilizing a microchip bumped with Cu-pillars. To determine the mechanical stability of the subjacent material stack, displacement-controlled Cu-pillar shear experiments were performed. To conduct the experiments, the sample and the AE sensor were placed in a customized holder system. The setup and the experimental approach are depicted in figure 1.

An advantage of AE measurements is the high acquisition rate which can reveal events not detectable with other methods. The mechanical data was acquired in this work with 2 kSamples/s and the AE signal with 100 MSamples/s.

Due to high sensitivity, AE measurements can also reveal minor damage events which cannot be identified by analyzing the mechanical data. The measured shear force and the AE signal of a Cu-pillar shear-off experiment are presented in figure 2.

Figure 2 b) shows the AE signal as well as the shear force progression over time in an interval of 25 ms around the shear-off event. The AE signal visualizes for example a small damage event before the main shear-off which is not indicated by the force measurement. The alignment of AE and mechanical data in this work was done manually, for further experiments an automatic feedback loop would be desired as presented in figure 3.

A feedback loop would enable a direct automatic correlation of the AE signal with mechanical data as well as the utilization of the acoustic signal to trigger actions of the indenter system. For example, an experiment could be stopped automatically as soon as an AE event is detected. The developed approach is not limited to shear experiments or semiconductor samples but can in principle be extended to any investigation in which structural damage is inflicted to a sample.

References:
10. In situ observation of dislocation evolution in cerium oxide nanocubes in an environmental TEM

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Cerium oxide, as one of the most important ceramic materials, is widely used in many applications, such as in solid oxide fuel cell electrodes, catalysis, or gas detection. However, there are few experimental pieces of evidence regarding the evolution of defects, which have a significant impact on the mechanical behavior.

Cerium oxides nanocubes (20-50 nm in size) are compressed using a dedicated Hysitron PI 95 sample holder in an environmental transmission electron microscope (ETEM). Plastic deformation of the nanocubes is analyzed using live High Resolution TEM imaging.

Cerium oxide nanocubes present a cubic structure (space group Fm-3m or Ia-3, depending on the gaseous environment and irradiation conditions). Their deformation mechanism is similar than that observed in other fluorite structures: when compressed along <001>, stacking faults are formed at higher stress by dissociation of <1 10> {1 1 1} dislocations. The dissociation of perfect dislocations is not a common phenomenon but has already been observed under electron beam irradiation [1, 2]. We will also discuss how nanotwins can be formed by multiplication of stacking faults [3].

References:

Abstract - PDF Poster Presentations

1. Strength of interface in bioresorbable poly-lactic acid/Mg fiber composites for orthopedic applications

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In the last decade researchers have shown interest in bioresorbable magnesium reinforced Poly-L-lactic Acid (PLA) composites to replace conventional non-degradable metallic implants in orthopedic application. Unfortunately, there is a lack of insight on the interfacial behavior between Mg and PLA which is necessary for optimal design of implant this material.

In this study, fabrication of bioresorbable composite of PLA reinforced with ultra-fine Mg (WE43) fibers of 100 microns in diameter is presented. Interfacial strength has been measured in their composite form exploiting a sophisticated and relatively accurate nano-indentation technique of push-out tests. The shear strength of the interface was measured by means of push-out tests in thin slices of the composite perpendicular to the fibers. It was found that the interface strength is $15.2 \pm 1.4$ MPa. While load-displacement behavior and SEM reveal nature of interface to be brittle. As the results of push-out test can be affected by constraining fibers, therefore non-isolated fibers were also pushed-out after random selection. It was found that constraint effect is negligible on the results of push-out test for Mg/PLA composites.
2. Screening of solvents for 2D materials exfoliation using nanoindentation

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The selection of appropriate solvents for the exfoliation of 2D materials from their bulk form mostly rests on semi-empirical correlations with surface energy terms [1],[2]. Herein, we present a method based on nanoindentation for the rapid screening and selection of solvents for the exfoliation of graphene and other 2D materials through measuring the first recorded pop-in on loading. The analysis of the load-displacement curves of nanoindentation on highly oriented polycrystalline graphite (HOPG) immersed in solvents show that the minimum average first pop-in depends on solvent composition. Figure 1a shows that with the solvent NMP (a standard exfoliation solvent) the mean stress for first popin is 4.58 ± 0.81 mN. Further investigation using solvent mixtures of isopropyl alcohol/water and ethanol/water at different ratios mix with water showed a minimum in the pop-in load occurring at or close to the solvent composition that showed the most effective exfoliation following ultrasonic bath sonication (figure 1-b,c). A similar correlation is seen between nanindentation of MoS2 in IPA/water mixture with the optimum composition at 40% IPA (figure 1-d).

References:

3. Acquisition of Residual Stress and Fracture Toughness of Metals Through Nanoindentation

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This study aimed to reveal the deformation induced mechanical properties of two advanced high strength steel grades, (namely DP 1000, and DP 1200); and an armour steel (Ramor 550) by means of nanoindentation and compare those with the non-deformed mechanical properties. To this goal, a Hysitron Ti950 equipped with Berkovich type indenter was used. Load controlled indentation with 6 mN maximum loading was performed on the samples. Hardness and modulus of elasticity maps as well as histograms were plotted and SEM, EBSD images of indents were obtained. Results showed a clear increased trend for hardness in some cases (e.g. DP 1000) and for modulus of elasticity for Ramor 550 armour steel. Indentation mapping exhibited strong correlation with the EBSD images for AHSS samples.
4. Single Crystal Thin Film Mechanical Behavior Measured with Nanoindentation

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One of the most common methods to measure the elastic modulus and hardness of thin films is to use nanoindentation and the well-known “10% rule of thumb” is utilized. The 10% rule of thumb has evolved to the understanding that elastic modulus and hardness can be taken at 10% of the film thickness with no or little influence from the substrate, even though only hardness was stated in the original Bückle paper. While this guideline may hold true for some film-substrate systems and film thicknesses (greater than 1000 nm), it cannot and should not, be applied universally. It will be shown on single crystalline copper films on sapphire, grown by thermal evaporation (50, 100, and 300 nm thick) that the hardness can be evaluated but the elastic modulus cannot be properly measured when compared to bulk single crystal copper. It will be demonstrated that the elastic modulus is a long range property that is substantially influenced by the substrate even at indentations of 10% of the thickness. For example, using the initial Hertzian elastic portion of the load-displacement curve before a pop-in occurs does not allow for the elastic modulus of copper to be measured. The findings reveal that the 10% rule should not be applied to evaluating the elastic modulus of thin films and hardness can be measured for some film system up to 50% of the film thickness.

5. In-situ electrochemical nanoindentation as a promising tool for probing hydrogen-material interaction

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In technical applications, materials often have to withstand harsh environments. Especially the exposure to hydrogen is a trouble causing issue in chemical industry and for energy storage applications involving hydrogen. Since high strength materials, such as high strength steels or nickel-base alloys, often suffer from significant susceptibility to hydrogen embrittlement, the investigation of mechanical properties under hydrogen influence is a promising field of research to understand the underlying hydrogen embrittlement mechanisms for further material development.

A convenient local characterization method is the electrochemical in-situ nanoindentation. Therefore, this in-operando nanoindentation technique was implemented in a G200 nanoindenter platform to study the nanomechanical characteristics of a nickel-base alloy under hydrogen charged condition. Beside reproducible measurements of standard mechanical properties, like hardness and Young’s modulus, deeper insight in the acting deformation processes can be gained by advanced testing methods. With nanoindentation strain-rate jump tests the determination of strain-rate sensitive properties on a local scale is viable. In combination with laser confocal microscopy the plastically deformed zone around the indents and the changes in slip step characteristics under hydrogen charging can be analysed.
6. Nanoindentation study of dislocation-based plasticity and crack formation in oxides

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In light of the rising topic of dislocation-based functionality of oxides, the dislocation-based mechanical behavior, for instance, dislocation plasticity and potentially crack formation induced by dislocations is also drawing increasing attention. Dislocations are line defects and the main carrier of plastic deformation. Understanding the dislocation-based mechanics in oxides can play a critical role in assessing the materials’ mechanical and operational reliability.

In this work, we present an approach to evaluate the incipient dislocation plasticity and crack formation using nanoindentation. In addition, the concept of “defect chemistry engineering” is proposed for the first time, based on which the dislocation plasticity of the oxide crystals can be tuned, as will be demonstrated in single-crystal SrTiO3. Two methods, i.e., via stoichiometry change of the Sr/Ti ratio, and by reduction treatment to increase oxygen vacancy concentration, have been validated to modify the dislocation nucleation and dislocation motion based on the nanoindentation study. These are both keys to dislocation plasticity. Furthermore, a unique size effect for brittle oxides, in comparison to metals, has been identified. Under a critical indenter tip radius, the plastic deformation in brittle oxides under the indenter tip will be purely governed by dislocations without crack formation.

7. The Effect of Loading Strain Rate on Nanoindentation Response of RTM6 Epoxy Polymer: Experimental and Computation Research

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In this paper nanoindentation response of a commercial epoxy resin (RTM6) is investigated by combining physical and computational experiments. A series of nanoindentation tests at different constant loading strain rates were carried out and corresponding mechanical parameters determined by the Oliver-Pharr approach. Since glassy polymers exhibit time-dependent yielding and knowing that hardness is directly related to yield stress, an increasing trend in indentation hardness with increasing strain rate would be expected. Conversely, experimental results show the opposite trend. In order to elucidate the cause of such apparent physically inconsistent results, finite element simulations experiments were performed. A nine-parameter elastic visco-plastic constitutive model (EVP-9) was calibrated from uniaxial stress-strain data available in literature and used to describe the visco-plastic nanoindentation response of the RTM6 epoxy system. It appears that inconsistencies in strain rate trends are due to the intrinsic visco-plastic nature of epoxy resin and not to spurious displacements measurements arisen from thermal drift effects. Our results undermining the appropriateness of Oliver-Pharr approach to investigate time-dependent properties like indentation hardness in glassy polymers.
8. **In-situ TEM investigation of toughening in Silicon at small scales**

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We present quantitative in-situ Transmission Electron Microscopy (TEM) fracture experiments on single crystal Silicon at room temperature. Findings consist of a brittle bulk fracture behavior of large samples at a stress intensity $K_{IC} \approx 1 \text{ MPa.m}^{1/2}$. However, below characteristic dimensions of about 250 nm, the fracture toughness strikingly increases inversely with size to at least triple.

Advanced in-situ TEM nanoscale strain mapping reveal the stresses at the crack tip approach the theoretical strength in small specimens. Strain mapping represents the tensile strain in the notch area in the opening direction of the crack, which reaches $\sim 8\%$ at a distance of $\sim 5 \text{ nm}$ from the notch tip in a specimen with thickness 136 nm thickness. For validation, FEM elastic tensile strain map of a computational digital twin with identical dimensions loaded to the same point is calculated and show excellent agreement and validating the plane strain condition and respective data analysis. Moreover, taking the average instantaneous force $F$ applied during strain mapping, the von Mises stress tensor, is calculated. Latter show that the Peierls stresses estimated as $[4.6–5.77] \text{ GPa}$ for Si are easily overpassed in the tip singularity region of small specimens. Thus, after dislocations nucleated, they can also propagate. TEM observations show that below this critical transition length, nucleation and propagation of dislocations occur, shielding the crack tip and enabling the unprecedented rise in fracture toughness.

9. **Fracture behavior of a tough bulk metallic glass at micrometer dimensions**

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Bulk metallic glasses (BMGs) are a promising material group for applications in micro- and nano-electro-mechanical-systems due to their unique physical properties, such as a comparably high hardness and elastic limit, paired with amorphous structure and certain glass specific traits. Despite significant research efforts performed on BMGs to date, spare data is available for mechanical properties at the micrometer size regime, as required for the above mentioned usage. While glasses commonly suffer from shear localization, leading to macroscopic brittle behavior, inherently ductile metallic glasses exist, which show extraordinary high resistance to crack propagation under bending load.

This special case of crack tip plasticity poses an issue to the conventional description of fracture toughness and therefore further detailed investigation on the fracture process is require. The present work strives for a deeper insight into the deformation mechanism and fracture behavior of a strong and ductile BMG, namely Pd$_{77}$Cu$_6$Si$_{16}$, in the micrometer regime. Multiple notched micro-mechanical samples in the shape of cantilevers were prepared by focused ion beam milling with different ligament sizes of 1, 2 and 5 μm, which were subsequently tested in-situ in a scanning electron microscope to observe the fracture process.

The specimens showed excessive blunting, which was captured by visual measurement of the crack tip opening displacement. Standardized fracture toughness models could not be applied due to a lack of significant crack propagation. Nevertheless, a significant size dependence in fracture behavior in the investigated regime was documented.
10. Probing structure-property relationships in Cu-Ni and Cu-Zn alloys in nanofoam form with nanoindentation

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The properties of nanostructured materials can differ from their equivalent bulk material by an order of magnitude. At the nanometer scale structural characteristics start to play an important role, and size effects can become the predominant factor on the final property. Using a novel polymer templating method, we fabricated low density (sub-10%) nanofoams of Cu-Ni alloys with different chemistries, ligament size and relative density. The modulus and strength was determined using nanoindentation tests to find the modulus and hardness of these materials. Relative density was found as one of the dominant parameters on the mechanical properties, showing significant decreases in strength and modulus around 6% relative density. The mechanical results are not only a consequence of the relative density; however, the combination of ligament size and solid solution strengthening do influence the result. The structure of nanofoams can be tailored to have desired outcomes by controlling the combination of foam chemistry, architecture, and density.

11. Mechanical property determination for cutting tool and workpiece using indentation and investigations on the boundary film formed in sliding contacts with emulsions

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Knowledge about the tribological interactions occurring between workpiece/chip and the cutting insert in lubricated turning processes is of great interest to model and to optimize the machining process. Taking the chip-tool contact as an example, the temperature at the interface can be easily over 400°C, which is usually much higher than that in lubricated contacts of machine elements. Before modeling the friction and heat partition of the contacts for a cutting process, as a primary step, it is essential to know the mechanical properties of the workpiece and cutting insert not only at room temperature but also under higher temperatures as the materials are exposed to in the cutting process.

To determine the Young’s modulus and hardness of the coated carbide cutting tool and the workpiece (42CrMo4), a Hysitron Ti 950 Nanoindenter with integrated heating stage was used. The measurements were performed with a Berkovich tip under room temperature, 300°C, and 400°C, respectively. It shows that the mechanical properties of the workpiece are significantly influenced by temperature, while the cutting insert does not change much till the tested temperatures.

Friction measurements have also been carried out in tribometer test, in which a ball is loaded against a steel plate in a heated lubricant tank doing reciprocating motion. The steel ball was coated in the same way as the cutting insert with AlTiN. The experiments were carried out mainly at boundary lubrication conditions in the presence of metalworking fluids. The formed boundary layer was investigated using nanoindentation. Thus it is possible to apply the determined mechanical properties of the respective partners into a simulation model for further analysis.
12. Micro-scale indentation on active microelectronic circuits to determine the piezoresistive coefficients of transistor channels

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Mobility shifts of the charge carriers in strained CMOS transistor channels occur due to changes of the silicon band structure under strain [1]. This effect is correlated with the used technology and the applied stress-strain fields. The piezoresistive coefficients of the channel material describe the sensitivity of the transistor channels to stress. The mentioned strain effect can be used to enhance the performance of transistors by including stressed layers but can also affect their functionality due to operational parasitic stress. Commonly, such effects are studied with four point bending setups enabling uniaxial stress and a direct feedback of the mobility shift vs. stress correlation [1]. A micro-mechanical test approach using non-destructive elastic indentation (Hysitron Ti 950) has been established to study these effects with high local resolution [2]. Particularly, such studies can be used to study localized stress related effects in microelectronic products e.g., due to chip-package-interaction. Prepared flip-chip-packages containing strain-sensitive ring oscillator (RO) circuits are used to monitor the circuit behavior during loading with spherical indenter geometries. The electrical responses of the circuits are compared with the mechanical stress-strain fields of the indentation contact using the finite element method (FEM). To determine the full set of directional stress-strain components and their influence on the RO behavior, different tip geometries for the experimental indentation setup have been selected using parametric FE studies. As a result, cylindrical tips have been introduced to selectively load the circuits [3]. The cylinder orientation according to the transistor channel direction is used to control the stress fields. Subsequently, a combination of three indentation experiments with fundamentally different stress-strain fields (spherical and cylindrical contacts in different orientations) is applied to determine the piezoresistive coefficients of the channel materials of the studied CMOS technology. Therefore, a linear independent set of equations connects the micro-indentation results e.g., RO behavior, the FE-obtained stress-strain fields and enables the determination of the unknown piezoresistive-coefficients, which are in good agreement with literature [1].

References:


13. Automated crack advancement detection for small scale fracture experiments

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Accurate knowledge of the current crack length is crucial for the evaluation of fracture mechanical tests. Macroscopically, this is achieved by well-established methods, e.g. the potential drop method, partial unloading technique or optical crack length observations. At the micron to sub-micron regime, such experiments are frequently performed in electron microscopes. There, the crack length is accessible via continuous stiffness measurements and/or image-based observations. The latter requires a high image acquisition rate to visualize crack growth. Digital image correlation, which is a standard method for image-based evaluation, can be hardly applied to in-situ crack growth experiments, as this technique requires a distinctly patterned surface. Hence, images are usually evaluated manually, which is a tedious and user-dependent task.

In this work, we performed miniaturized in situ fracture experiments on focused ion beam fabricated TiAl alloys within a scanning electron microscope using a Hysitron PI 85 nanoindenter equipped with a continuous stiffness measurement module. Image processing techniques are subsequently utilized in order to introduce a semi-automatic procedure to measure the crack extension and improve the crucial step of crack growth measurement. The semi-automatic procedure searches for contour lines around the previous crack shape and locates the new crack tip according to the position on the previous frame. The crack length results obtained by the semi-automatic procedure agree well with the manual assessment as well as the crack length determination using the continuous stiffness data. Thus, our semi-automatic procedure enables the investigation of small-scale fracture processes in more detail, as a more data can be analysed user-independent, which in particular benefits detection of slight crack growth events.
14. Using machine learning algorithms to interpret finite element simulations of indentation experiments including tip radii effects

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Machine learning algorithms have already been used to interpret indentation data. In this study, different machine learning models will be used on data with different fidelities, closing the gap from 2D and 3D simulations. This will be done using Residual Multi Fidelity Neural Networks and other machine learning techniques. Data gained by finite element simulations of the indentation process will be used to find features in indentation curves and train machine learning algorithms for predicting elasto-plastic parameters and tip radii. The estimation of tip radii is expected to enable insights into tip wear during indentation experiments in future.

15. Fatigue Tests of Penta-twinned Ag Nanowires under TEM and Their Structure Analysis

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Ag nanowires (NWs) have applications in flexible electronics because of their excellent electrical and optical properties. The polyol process used to fabricate Ag NWs leads to a distinctive penta-twinned structure containing five {1 1 1} twin planes sharing a common axis along [1 10]. Here we study the mechanical performances of these wires through TEM analysis of individual NWs after cyclic deformation. This is achieved by spraying Ag NWs onto 3mm porous polycarbonate disks covered by an electron transparent collodion thin film, selected fibres suspended over pores in the disks can be identified for repeated TEM study after deformation. The disks are repeatedly cycled from 0 – 8% tensile strain and individual NWs are selected and characterized before and after fatigue tests. A bamboo defect structure, defined as repeated narrow regions of contrast difference across the diameter of the penta-twinned Ag wires that repeat along a wire at distances significantly greater than the wire diameter, is observed under TEM observation. An increase in density of bamboo defects in the Ag NW networks is observed after increasing numbers of fatigue cycles. Further characterization using precession assisted scanning nanobeam electron diffraction (NBED) suggests that the bamboo structure is caused by crystal rotation in the penta-twinned NWs around the [1 10] growth direction. We propose that the torque that generates rotation is induced by the presence of NW/NW joints within the network allowing circumferential loading of individual NWs when the network is in global tension. Fewer bamboo structures are observed after fatigue when examining lower density NW networks, supporting the network joint hypothesis.