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FACULTY OF NUCLEAR SCIENCES AND PHYSICAL ENGINEERING CTU IN PRAGUE

BOOK OF ABSTRACTS

LOCAL MECHANICAL PROPERTIES 2024

16TH INTERNATIONAL CONFERENCE ON LOCAL MECHANICAL PROPERTIES

PRAGUE, CZECH REPUBLIC

29-31 MAY 2024

LOCAL MECHANICAL PROPERTIES 2024

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9:00-12:00	MecaNano workshop	for registered participants (MecaNano members)

Wednesday 29 May afternoon – MecaNano special section

12:00-13:45	*Registration*	
13:45	*Opening*	
Properties at micro/nano scale		
14:00	Marc Legros *(invited)*	Micromechanical Assessment of Fracture Properties of Austenitic Stainless Steel Grain Boundaries Oxidized in a Pressurized Water Reactor Environment
14:40	Szilvia Kalacska *(keynote)*	Irreversible evolution of dislocation pile-ups during cyclic microcantilever bending
15:10	Stanislav Žák	Nanoindentation with sub-surface particle sensing
15:30	Ronan Henry	Multi-scale fracture testing of Ni-20Cr alloy printed by Laser Power Bed Fusion
15:50	*Coffee break*	

Nanoindentation challenges		
16:15	Megan Cordill *(keynote)*	A shallow jump in to how nanoindentation can be used effectively
16:45	Jiri Nohava	Investigation of shape and area function of indenters in various states
17:05	Yvan Marthouret	Indentation of rough surfaces
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18:05		Alemnis
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Thursday 30 May morning

Properties of surfaces, layers and particles I		
8:30	Andreas Mortensen *(keynote)*	Local mechanical properties of oxide inclusions
9:00	Sandor Lipcsei	Micromechanical properties of MnO-SiO_2-Al_2O_3 inclusions in iron
9:20	Davide Vacirca	Boosting mechanical properties of thin film high entropy alloys through nanoengineering design strategies
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10:00	Šárka Houdková	Wear behavior of selected HVOF sprayed WC-Cr_3C_2-M coatings under different types of mechanical loading
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Properties of surfaces, layers and particles II		
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11:10	Frantisek Lofaj	The comparison of micromechanical properties of the compositionally complex transition metal nitride coatings deposited by different reactive sputtering techniques
11:30	Dávid Ugi	Plastic instabilities and strain recovery in amorphous LiPON thin layer
11:50	Thomas Lindner	Surface functionalization in selective laser melted 17-4 PH by plasma polishing and interstitial diffusion hardening
12:10	Aliasghar Najafzadehkhoee	Contact damage of multi-material laminar ceramics: effect of layers architecture and residual stress
12:45	***Photo***	
13:00	*Lunch*	

Micromechanical testing of irradiated concrete		
11:10	Jiří Němeček *(keynote)*	Fracture properties of Calcium-Silicate-Hydrates exposed to γ-irradiation and different relative humidities
11:40	Jiří Němeček	Micromechanical characterization of γ-irradiated cement paste exposed to different relative humidity conditions
12:00	Jan Procházka	Optimization of FIB milling procedure for micromechanical testing of cement pastes
12:20	Ondřej Libera	Nanomechanical properties of neutron irradiated concrete

Thursday 30 May afternoon

Experimental challenges and coupled methods I		
14:20	Benoit Merle *(invited)*	High strain rate persistence of the strength anomaly in L12 intermetallic compound Ni3Si evidenced by nanoindentation at elevated temperatures
15:00	Mohammed Tahir Abba	Experimental Progress in High Constant Strain Rate Nanoindentation
15:20	Thomas Chudoba	Fatigue testing of coatings by using the dynamic capabilities of a nanoindenter
15:40	Marcel Sos	Nanoindentation methods for analysis of thermally activated processes at elevated temperatures
16:00	Renato Pero	High Strain Rate Nanoindentation – Recent Advances and Perspectives
16:20	*Coffee break*	

Microeletronic materials		
15:10	Kiran Mangalampalli *(keynote)*	On the phase-engineered novel phase of Silicon
15:40	Swarnendu Banerjee	3D Tomography on Advanced Photovoltaic (PV) Structures – Examples of Good Practice
16:00	Sergio Sao Joao	Multi scale in-situ micromechanical testing of Polymer Core Solder Ball (PCSB) coatings for BGA interconnections

Experimental challenges and coupled methods II		
16:50	Péter Dusán Ispánovity	Unraveling acoustic emission by coupling it to micromechanics
17:10	Antoine Ollivier	High-resolution measurement of strain by tracking of nanoparticles
17:30	sponsors	Anton Paar
17:50		Keyence
18:10		Jeol
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Friday 31 May morning

Micromechanical testing of polymers		
8:30	Jaroslav Lukes	Comparison of Spherical Indentation Analysis for Soft Polymers in Bio Applications
8:50	Miroslav Šlouf	Macro-, micro- and nanomechanical characterization of crosslinked polymers with very broad range of mechanical properties
9:10	Veronika Gajdošová	Comparison of macroscale, microscale and nanoscale creep behavior of UHMWPE and PEEK polymers used in total joint replacements
9:30	Marina Melo de Lima	Comparison of Berkovich and spherical tip indentation for determining the Young's modulus of polymer thin films encapsulated by a dielectric capping
9:50	*Coffee break*	

Challenging materials		
10:20	Tamás Csanádi	The effect of crystal anisotropy on fracture toughness and strength of ZrB2 microcantilevers
10:40	Ali Talimian	Scratch behavior of chemically tempered alkali borosilicate glass: measurements using Berkovich and conical indenter
11:00	Pavol Hvizdoš	Nanohardness, fracture resistance, and enhanced wear resistance of dual-phase high-entropy carbide/boride ceramics
11:20	B. Erdem Alaca	Understanding Intrinsic Stress Effects on Vibrational Response of Silicon Nanowires
11:40	Krishna Sarath Kumar Busi	Understanding and validating the fracture behavior of damage tolerant diamond-metal laminates
12:00	*Closing*	

Posters

Elisaveta Georgieva Kirilova	Multi-parameter optimization of layered WS2-polymer nanocomposite under mechanical loading
Rayka Kirilova Vladova	Parametric analysis for interface shear stress in MoS2/PET nanocomposite under thermo-mechanical loading
Magdalena Mrózek	Addition of dental fillings with nanoparticles to improve their mechanical properties
Josef Daniel	Effect of selected HVOF-sprayed coatings parameters on dynamic impact wear
Maroš Martinkovič	Utilization of the Shear Test to measure the Local Mechanical Properties of Duplex Steels Welds
Pavel Kovačócy	Influence of Laser Beam Welding Parameters on Local Mechanical Properties of Duplex Stainless Steel Joints
Bruno Passilly	About the choice of the indenter to determine mechanical properties of superalloys by using high temperature microhardness tester
Radek Šlesinger	Efficient mapping of mechanical properties using Gaussian processes
Michael Huszar	Modulus estimation of polymers via nanoindentation – impact of surface roughness and peak force
Matej Kubiš	Nanoindentation analysis of high fluence helium ion irradiated Eurofer 97 and ODS Eurofer steels
Wojciech Żórawski	Mechanical Properties of Cold-Sprayed Ti-6Al-4V Coatings on Al 7075 Alloy
Jan Tomáštik	A nanoindentation study of TiN films deposited using magnetron sputtering under various condition
Medard Makrenek	A new approach to the properties of matter in terms of nanoindentation research on the example of selected materials
Jadranka Blazhevska Gilev	Mechanical and thermal reinforcement of the polymer based graphene nanoribbon composites
Michal Krafka	Changing the integrity of the material surface by combining Laser Surface Texturing and PVD Magnetron Sputtering technologies
Jaroslav Kovář	Finite element analysis of the pile-up and correction of projected contact area
Wiktor Bednarczyk	Solid solution strengthening of Zn-based alloys measured by micro-pillar compression
Petra Christöfl	Nanoindentation creep behavior of UHMWPE and PEEK polymers used in total joint replacements
Aleš Materna	Estimation of Mechanical Properties of Neutron-Irradiated 08Ch18N10T Steel from Hardness Testing
Vojtěch Schánilec	AFM-in-SEM: Understanding mechanical properties of low dimensional materials
Zoltán Száraz	Nano- and micromechanical testing of helium implanted reactor materials

Properties at micro/nano scale

Micromechanical Assessment of Fracture Properties of Austenitic Stainless Steel Grain Boundaries Oxidized in a Pressurized Water Reactor Environment

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Keywords: Micropillar compression and microbeam bending, nuclear environment Stainless steel, grain boundaries, oxydation

Austenitic stainless steels are extensively utilized in aqueous environments owing to their robust corrosion resistance. However, the emergence of Inter Granular Stress Corrosion Cracking (IGSCC), notably within primary circuits and internal structures of Pressurized Water Reactors (PWRs), poses significant challenges. Particularly in the case of 304/316 steels, this degradation phenomenon is referred to as Irradiation-Assisted Stress Corrosion Cracking (IASCC). Understanding the fracture properties of austenitic stainless steel grain boundaries (GBs) oxidized in PWR environments is thus imperative for predictive modelling of IGSCC.

In this study, we assess the fracture properties of oxidized FeCr12Ni26Si3 (wt %) austenitic stainless steel GBs in a PWR environment. The alloy's chemical composition closely mimics that of 304/316 stainless steels' GBs after irradiation, rendering the obtained results relevant for IASCC. Micro-cantilever beams containing a single grain boundary were milled on oxidized samples using Focused Ion Beam (FIB) and subjected to in-situ SEM testing at room temperature. Intergranular cracking was observed in 12 micro-beams, occurring either within the GB oxide or at the oxide/metal interface. Initially, the Coupled Criterion (CC) theoretical framework for crack nucleation was employed to estimate the fracture properties. These estimates were then refined by comparing experimental results to Finite Element (FE) simulations using the Cohesive Zone Model (CZM).

Additionally, micropillar testing was performed to assess the yield stress and hardening behavior of the steel used in the micro-beam experiments.

A notable agreement was achieved for the macroscopic fracture load, with a fracture energy (γc) approximately equal to 11 ± 3 J.m−2 and a strength (σc) of oxide around 1000 ± 250 MPa. These values were subsequently compared to existing literature results and critically evaluated regarding their dependence on experimental and numerical parameters.

Irreversible evolution of dislocation pile-ups during cyclic microcantilever bending

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Keywords: micromechanical testing, cantilever bending, copper single crystal, HR-EBSD, GND density

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].

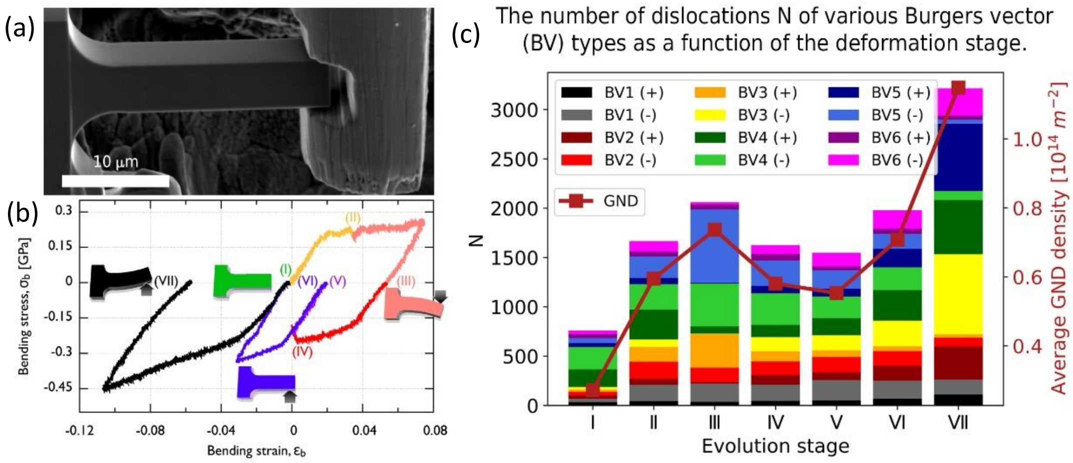

Fig. 1: (a) Secondary electron image of the deformed cantilever, (b) Bending stress-bending strain curve. The bending experiment was performed in 6 steps (indicated by the different colours). HR-EBSD scans were conducted at the initial state and after each step, as indicated by the roman numerals (I)–(VII). The four sketches illustrating the state of the cantilever are to help the interpretation of the figure and the arrows represent the bending direction prior to the actual measurement. The number of dislocations N of various Burgers vector (BV) types as a function of the deformation stage.

References

[1] D. Ugi *et al.*, Irreversible evolution of dislocation pile-ups during cyclic microcantilever bending. (2023) DOI: 10.48550/arXiv.2311.14018

Nanoindentation with sub-surface particle sensing

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Keywords: elastic-plastic strain field, finite element method, inverse analysis, nanoindentation, steel

In order to improve manufacturing processes, there is a high demand on development of new high-strength materials. These materials have to satisfy the need for not only strength, but also a fatigue reliability. One way to improve the fatigue and crack propagation resistance is the directed microstructure engineering. In order to assess the fatigue resistance of any material, its mechanical properties must be known and the influence on any microstructural changes on them must be assessed. A suitable way of measuring the elastic and plastic material properties on a micro-scale is with nanoindentation. However, a microstructure containing hard carbides embedded in softer matrix present an interesting challenge for nanoindentation, especially sensing hard particles under the surface, thus compromising the results by unknown influence. To investigate such phenomena, nanoindentation was performed on two chemically same, but microstructurally different steel samples. It was complemented by the numerical simulations, revealing the high influence of carbides under the surface on the matrix indents. Presented results show large extent of elastic and plastic zones under the indenter tip. Additionally, the direct influence by hard carbide particles with different sizes and compositions on the deformation field is examined. The combination of experiments and numerical simulations provide a clearer understanding of the measured mechanical response of high strength steels that are normally considered "simple" at the macro-scale, but in reality, are quite complex at the micro- and nano-scales.

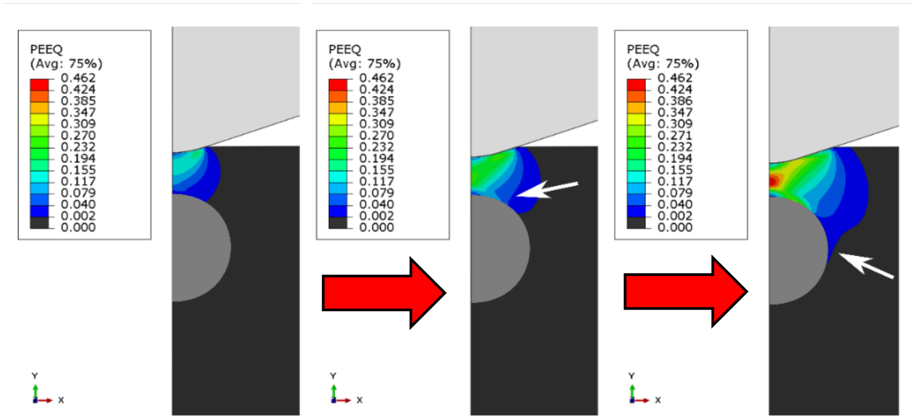

Fig. 1: Plastic zone development under the indenter tip with a hard particle throughout the nanoindentation, shallow indent on the left, deep indent on the right, white arrows indicate plastic zone sticking to the carbide.

Acknowledgement

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Multi-scale fracture testing of Ni-20Cr alloy printed by Laser Power Bed Fusion

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Keywords: Fracture toughness, size effect, ductility, bending test

The fracture behavior of a Ni-20 wt%Cr binary alloy manufactured by Laser Powder Bed Fusion (LPBF) is studied at difference scales by performing bending tests on notched specimens.

Micro-bending toughness tests are important to test small components (MEMS, coatings, microstructure elements, etc.) [1]. This type of test is classically affected by size effects on brittle materials, but is very much affected by size effects on ductile materials [2].

With the aim of studying the measurement of toughness on ductile materials at different scales, different sizes of notched cantilevers are prepared and tested. Macroscopic tests consist of three-point bending (Fig. 1: a,d) conducted on specimens of a few tens of millimeters. The mesoscopic scale characterization relies on tests performed on cantilevers of a few tens of microns machined with plasma FIB (Fig. 1: b,e). Finally, microscopic testing deals with bending tests carried out on cantilevers of a few tens of microns machined with Gallium FIB (Fig. 1: c,f). These last two configurations were tested by means of an *in situ* nano-indenter integrated in a Scanning Electron Microscope. In all cases, crack initiation and propagation are monitored during the test by imaging, and the fracture toughness results are compared and discussed as a function of the specimens' size along with their specific microstructure and their characteristic internal length.

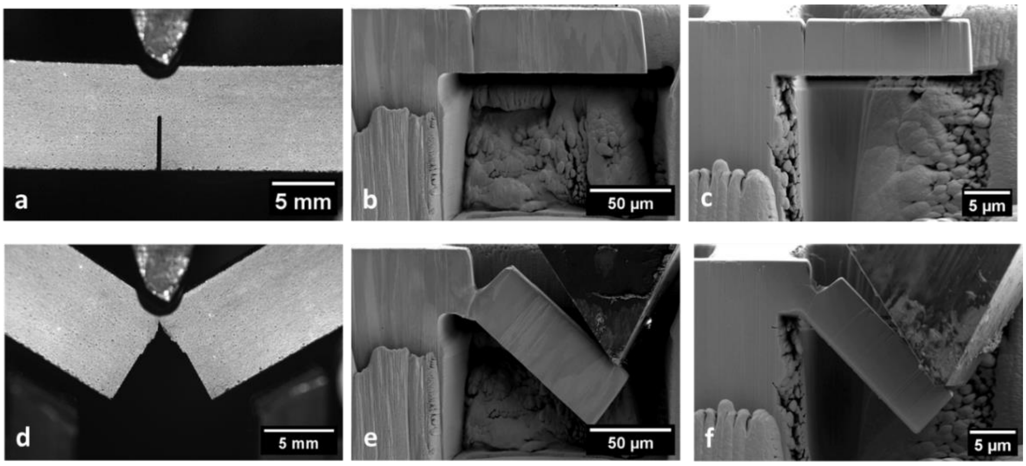

Fig. 1: In situ fracture testing of Ni20Cr alloy bending specimens: Initial state (a,b,c) and fractured state (d,e,f) macroscopic (a and d), mesoscopic (b and e) and microscopic (c and f) .

Acknowledgement

The authors would like to acknowledge the Labex EMC3 for the MIFASOL project funding.

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Nanoindentation challenges

A shallow jump in to how nanoindentation can be used effectively

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Keywords: steel, nanoindentation, pop-in behavior, finite element method, inverse analysis

Since its introduction in the early 1990s, nanoindentation has become a popular and useful technique to measure mechanical behavior, especially of small volumes. Because of nanoindentation's popularity and ease of use, nanoindentation became the "It Girl" for thin film mechanical behavior and eventually the emerging field of nano- and micromechanics. The technique and instrumentation have also evolved over the past 30 years to offer high/low temperatures, displacement/load control, quasi-static/dynamic indentation, mapping, and scanning imaging. From the methodology and instrumentation advances, the community moved forward rapidly – especially with respect to using a nanoindenter in the nano- and micromechanics fields. What the community may be missing is the return to the past and the use of the new and advanced tools available to address the early questions of tip wear, indentation size effects, and measuring the elastic modulus of thin films. Through the application of finite element analysis, self-imaging of tips, and machine learning, these early topics will be re-examined on a variety of material systems to provide a more thorough understanding of what the nanoindentation community thought it knew. The end goal is to better educate the next generation to apply nanoindentation methods more effectively.

Investigation of shape and area function of indenters in various states

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Keywords: Berkovich indenter, area function, blunting, tip radius

Correct calculation of H_{IT} and E_{IT} relies on nanoindentation instrument's force and depth calibration, thermal stability, frame compliance and indenter area function. While force and depth calibration and frame compliance are factory calibrated and good thermal stability achieved by stabilization time or instrument design, indenter area function has to be determined for each indenter regularly during its use. This is crucial especially at low depths (<200 nm) where blunting of the indenter plays an important role. ISO 14577 recommends for nanoindentation indenters with tip radius smaller than 200 nm. However, in real life it is very difficult to measure directly the tip radius and vast majority of users rely on indirect methods based on indentation of certified materials. Our work investigates several approaches that allow for estimation of tip radius and for determination of the usable depth range for reliable hardness measurement. A large part of our work is based on an analysis of several tens of Berkovich indenters by indentation on fused silica. By fitting a spheroconical model to the projected area (A_p) data we were able to estimate the tip radius and the blunting depth h_b which, together with the spherical depth h_{sp} provide a good approximation of tip blunting. The so-obtained tip radius was verified on several indenters using an AFM measurement. It was found that even a direct measurement of the indenter tip radius suffers of up to 50 nm uncertainty. However, since the spheroconical model is a simplification of the real shape, we plotted also $\sqrt{A_p}$ (obtained by indentation of fused silica) as a function of contact depth h_c and compared it with the $\sqrt{A_p}$ of an ideal indenter. The depth h_T at which the $\sqrt{A_p}$ curve becomes parallel to that of the ideal indenter is related to the depth at which the indenter becomes conical and can be taken as a minimum depth for reliable hardness measurement. Finally, hardness of fused silica at various depths obtained with indenters whose shape changed during their use was compared. The hardness was calculated using original A_p (without damage) and A_p of the damaged indenter. We have observed that for severely damaged indenters a recalibration of A_p does not provide reliable hardness values (Fig. 1). We believe that the above-mentioned approaches for estimation of indenter blunting and usable depth for reliable hardness measurement will contribute to more reliable analysis of nanoindentation measurements.

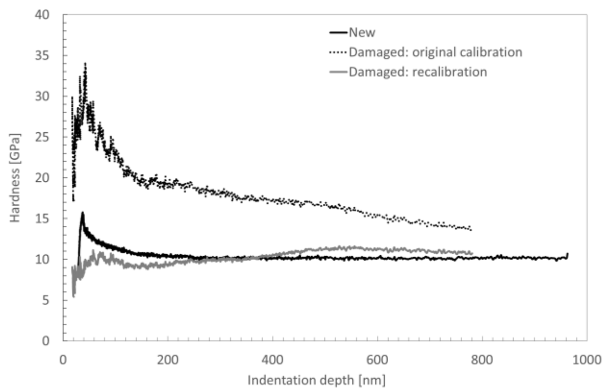

Fig. 1: Hardness of fused silica as a function of depth obtained using new and damaged indenter with different area functions.

Indentation of rough surfaces

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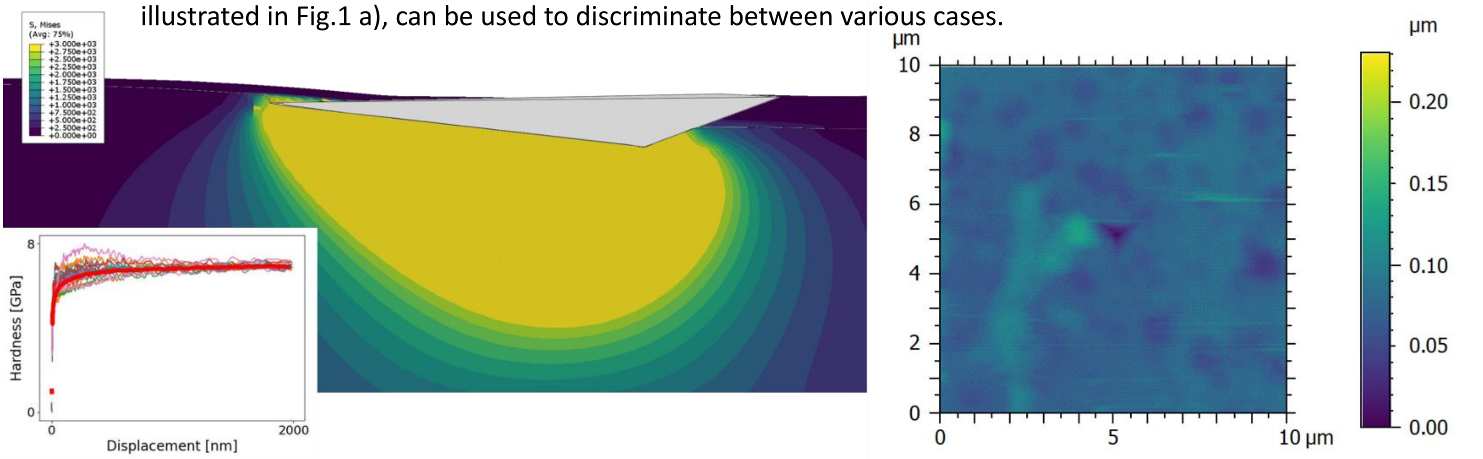
Keywords: nanoindentation, roughness, finite element method, inverse analysis

Nanoindentation is usually performed on polished samples in order to process data with traditional models [1] and to avoid large discrepancies arising from roughness. However, polishing is sometimes not possible: for example when studying very thin coatings or samples with a surface treatment [2]. As shown in Fig. 1 a) insert and described in [3], the mechanical properties measured on rough surfaces exhibit a large dispersion. One can then ask whether this dispersion is due to the morphology, to an inappropriate model or to a gradient in surface properties induced by machining. In the latter case, it could mean that close surfaces (mainly local asperities) may have different properties than the bulk. In this regard, Fig.1 b) shows a topographic image of an indent with its surrounding surface, measured *in situ* by the indenter. The relationship between mechanical properties and morphology can thus be established. Finally, a comparison with finite element method simulation of the indentation, as illustrated in Fig.1 a), can be used to discriminate between various cases.



a) b)

Fig. 1: a) Finite element simulation demonstrating the indentation of a rough surface by a Berkovich tip,
Insert: hardness evolution as a function of depth carried out on rough surface
b) Imprint on rough surface, measured by indenter tip in SPM mode

Acknowledgement

This work was carried out within the MesuRufo project (Chaire de Professeur Junior, cofunded by ANR).

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Recent innovation in Scanning Electron Microscope (SEM) in-situ extreme mechanics at the micro and nanoscale

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Keywords: In-situ, High Temperature, Low Temperature, Micromechanical Testing, High Strain Rate

Nanomechanical tests are moving beyond the basic measurement of hardness and elastic modulus to encompass a host of different mechanical properties such as strain-rate sensitivity, creep, and fracture-toughness by taking advantage of focused-ion beam milled geometries. New developments, such as high-cycle fatigue, are extending the range of properties which can be studied at the micro and nanoscale. However, such techniques are challenging due to low oscillation frequencies, long duration of tests and large thermal drift when attempted with standard indentation instruments. Novel piezo-based nanoindentation methods are now allowing access to extremely high-strain rates (>10^4/s) and high-oscillation frequencies (up to 10kHz).

Until only recently, high-strain rate (10^2/s-10^4/s) testing of materials has only been possible using macroscale techniques, *e.g.*, split-Hopkinson bar, Kolsky bar and plate-impact tester. At the microscale, strain-rates have typically been limited to ~10^{-1}/s or less, owing to limitations in instrumentation, insufficient data acquisition rates and elastic wave propagation conflicts during testing.

This talk will focus on the most recent developments in instrumentation for in-situ extreme mechanics testing at the micro and nanoscales, with specific focus on a testing platform capable of strain rate testing over the range 10^{-4}/s-10^4/s (8 orders of magnitude) with simultaneous high-speed actuation and sensing capabilities, with nanometer and micronewton resolution. A selection of case studies will be presented on topics of interest, including, crack propagation, scratch testing, cyclic loading (tensile and compression), tribology, fretting, impact, fracture toughness and combination of variable temperature (from -150 to 1000°C) and variable strain-rate testing. Finally, future research directions in this sub-field of micromechanics will be discussed.

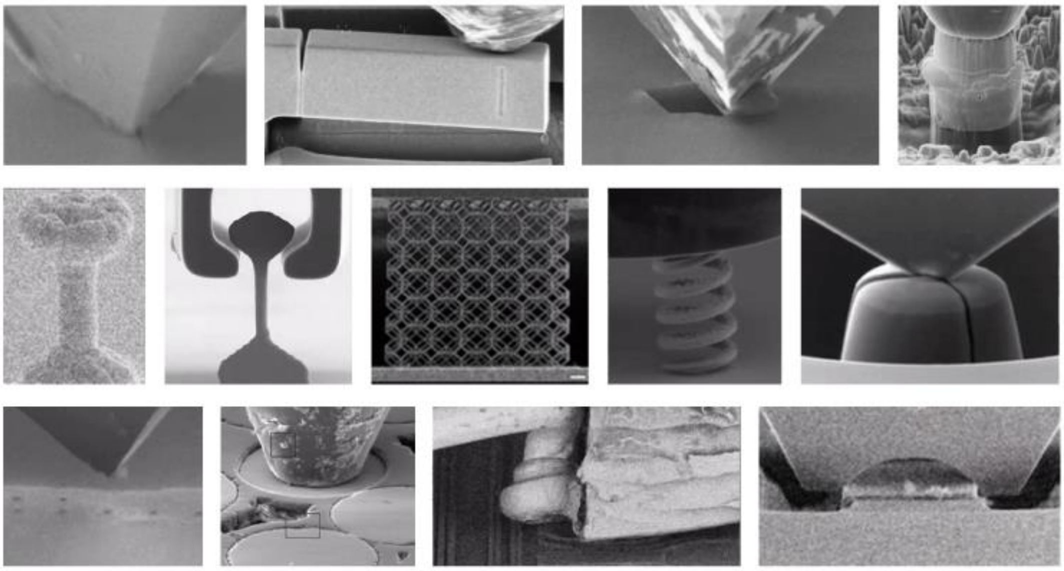

Fig. 1: Typical micromechanical testing methods performed in-situ in SEM.

Properties of surfaces, layers and particles

Local mechanical properties of oxide inclusions

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

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Keywords: inclusions, oxide, steel, nanoindentation

Oxide inclusions, which are practically inevitable given the need for deoxidation during steelmaking, are known to affect 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, *in-situ*, mechanical properties of small objects such as discrete micron-scale second phases in alloys has seen significant progress over the past decade. This motivates the present exploration of the link between inclusion composition and inclusion mechanical properties, with a goal to identify inclusion compositions that have minimal negative impact on the mechanical performance of steel. An adaptation of the Oliver-Pharr method is proposed to measure, via nanoindentation, the hardness and stiffness of inclusions embedded in a matrix that has different micromechanical properties. Using the proposed indentation procedure, we explore the stiffness and hardness of inclusions belonging to the $SiO_2 – Al_2O_3 – CaO$ system; this includes notably calcium aluminates that are expected upon successful calcium treatment of molten steel to prevent solid alumina inclusions from clogging nozzles during continuous casting.

Acknowledgement

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Micromechanical properties of MnO-SiO_2-Al_2O_3 inclusions in iron

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Keywords: inclusions, oxide, steel, nanoindentation

Oxide inclusions in the SiO_2 – Al_2O_3 – MnO pseudo-ternary system are precipitated within iron by *in-situ* reaction, analyzed for their composition and characterised by means of micromechanical tests that give their intrinsic, *in-situ* mechanical properties such as their hardness and their stiffness, or the strength of their interface with the iron-based matrix. Preliminary results suggest that adjusting the MnO content of the resulting oxides inclusions can be used to obtain an inclusion stiffness close to that of the iron matrix, thus reducing the elastic inclusion/matrix mismatch in steel and making MnO-rich inclusion compositions attractive candidates if one seeks to minimize local stress concentrations in the alloy under mechanical loading.

Acknowledgement

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Boosting mechanical properties of thin film high entropy alloys through nanoengineering design strategies

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Keywords: high entropy alloys, thin films, nanocrystalline, nanolaminates, micropillar compression

Thin film high entropy alloys (TF-HEAs) are gaining attention for their large ductility and yield strength, as a result of their small grain size and the activation of mechanical size effects [1]. Moreover, hardness and yield strength can be improved by developing nanolaminate structures, capable of blocking dislocations and crack propagation at the interfaces [2]. However, developing new nanoarchitectures and investigating local mechanical properties using *in situ* SEM techniques is still an open challenge.

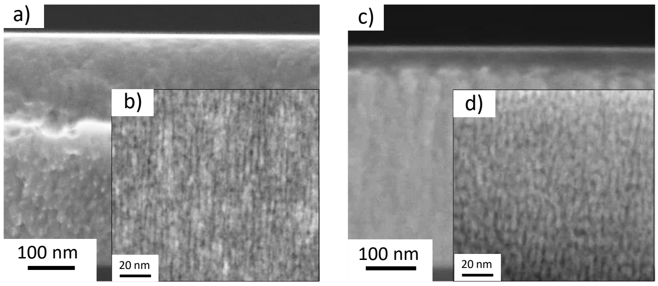

Figure 1 - SEM and STEM (inset) cross sections of compact (a,b) and nanogranular (c,d) CoCrCuFeNi

In this work, we developed nanoarchitectured TF-HEAs by magnetron sputtering and pulsed laser deposition (PLD) in combination with thermal annealing treatments, enabling a fine control over the film's morphology while also developing nanolaminated structures with precise control of the interface density.

Firstly, we focus on the synthesis of nanostructured CoCrCuFeNi TF-HEAs by PLD. We report a transition compact → nanogranular for a background pressure >1 Pa (Fig.1), resulting in a mild density decrement due to cluster-assembled growth (down to 6.91 g/cm^3). Moreover, these films show greater hardness (10.5 GPa) compared to magnetron sputtering (7.4 GPa), while showing exceptional ductility in tensile tests on polymer substrate (onset of crack formation at ε=3.4%). Thermal annealing (investigated by *in situ* XRD) shows grain coarsening starting from 400°C and the formation of a $FeCu_3$ phase, resulting in H decrement (down to 8.5 GPa).

Secondly, we fabricated Al/HEA nanolaminates with bilayer period (Λ) ranging from 2.5 to 200 nm. Among the main results, we show that Al/CoCrCuFeNi nanolaminates (semicoherent, FCC/FCC) maintain high hardness, up to 9.7 GPa (Λ=50 nm) despite a volume fraction of 50% for Al (H≈1.5 GPa), while $Al/Al_{25}(CoCrCuFeNi)_{75}$ (incoherent, FCC/BCC) show high ductility (no brittle fractures at 30% deformation) and a yield strength of 2.5 GPa (Fig. 2).

Overall, our results show how a nano-engineer design of TF-HEAs results in improved and tunable mechanical properties with key implications for industry applications.

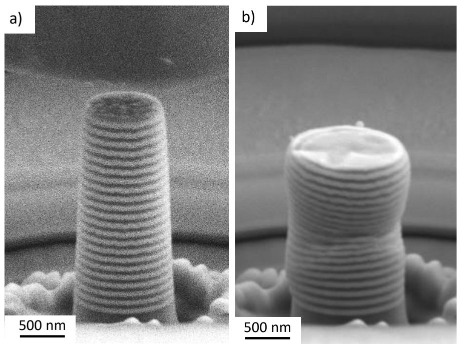

Figure 2 – Micropillar of FCC/BCC multilayer (Λ = 100 nm) before (a) and after (b) compression up to

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Local Cohesion of Splats in Hybrid Plasma Spray Coating as Observed by In-Situ Experiment

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Keywords: plasma spraying, hybrid coating, in-situ SEM, strain mapping, cohesion

Hybrid plasma spraying combines spraying of dry coarse powders and liquids (suspensions and/or solutions). Introduction of secondary miniature phase from the liquid may provide new coating functionality but also may have a significant influence on the local coating cohesion – positive or negative, depending on the materials combination and deposition conditions. In this study, cohesion of dissimilar splats within the hybrid coating will be evaluated by in-situ SEM observation during bending and results will be related to the macroscopic properties of the coating and its previous thermal history.

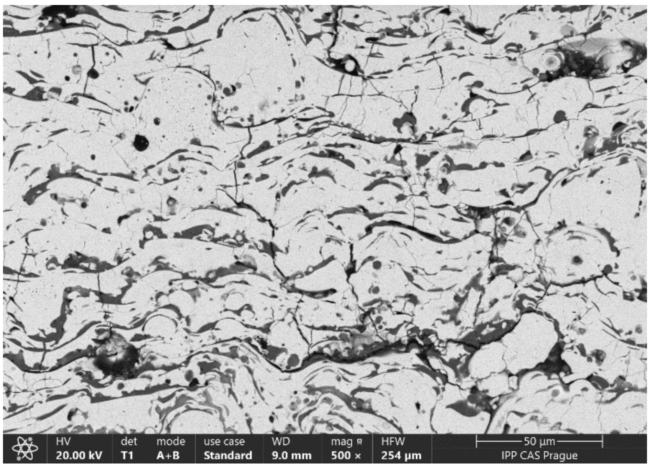

Fig. 1: Heterogenous microstructure of hybrid YSZ-Al_2O_3 coating showing YSZ (bright phase), miniature Al_2O_3 (dark phase) and voids. Cross-section, SEM.

Acknowledgement

Financial support through project 22-21478S "*High-enthalpy deposition of hybrid plasma spray coatings*" funded by Czech Science Foundation is gratefully acknowledged.

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Wear behavior of selected HVOF sprayed WC-Cr_3C_2-M coatings under different types of mechanical loading

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Keywords: HVOF, WC-Cr_3C_2-Ni, abrasion, erosion, wear, dynamic impact test

The wear resistance is one of the most frequently required functionalities on the surface treatment. Among thermally sprayed coatings, the hardmetal coatings are generally considered to provide the optimal combination of hardness and toughness, leading to a high wear resistance. The mutual ratio between the amount of hard phase and metal matrix and their composition can be decisive with regard to different types of loading. The work is focused on the comparative evaluation of WC- Cr_3C_2-M HVOF sprayed coatings with various amount of WC, Cr_3C_2 and metal matrix, including two commercially available compositions, as well as a newly designed one, in terms of microstructure, mechanical and tribological properties. As a benchmark, the results of the most frequently used WC-12CoCr (WOKA 3652) and Cr_3C_2-25NiCr (Amperit 588) are also presented. The abrasive wear and solid particle wear resistance was related to the results of dynamic impact wear resistance test. Although the hardness of all the evaluated coatings is very similar and can be related to a similar abrasive wear behavior, their solid particle erosion wear and dynamic impact wear resistance differ significantly as a result of differences in the metal matrix amount and composition.

Acknowledgement

This work was performed under the support of the project "Development of "3D print-thermal spray" systems for applications with dynamic and impact loading (DePriSS)" of the M-Era.Net program. It is co-funded by the Free State of Saxony and the Czech Republic. In Saxony it is supported under contract 100575222 via SAB (Saxonian Aufbaubank) from tax money based on a decision of the Saxonian Parliament. In the Czech Republic the project was supported by the Technology Agency of the Czech Republic, under contract no. TH75020003.

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On the use of nanomechanical testing to characterize transformations of materials induced by surface manufacturing processes

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Keywords: Tribologically Transformed Surfaces, microcompression, nanoindentation, temperature, microstructure, thermal stability

Surface manufacturing processes - such as machining, shot peening, burnishing, polishing … - are known for their consequences on surface integrity. They are mostly triggered by repeated and intense contact loadings leading to large plastic deformation, high strain strain rate and high temperature rise in the near-surface [1]. A significant in-depth gradient of mechanical properties is usually observed over 10 to 100 µm depending on the process and is usually named “Tribologically Transformed Surface”. The TTS is a consequence of near-surface materials transformation and can play on materials performance (fatigue, stress-corrosion, wear). The accurate characterization of the mechanical properties of these new materials at the right scale is therefore of primary importance. It can be made through the use of suitable methodologies based on nanomechanical testing –i.e. micropillar compression, nanoindentation. The nanomechanical testing field is actually reaching a maturity level that allows its deployment to materials transformation induced by surface manufacturing processes (Fig 1). The first part of this presentation will be dedicated to a brief review of the last developments in the nanomechanical testing field, with a special focus on extreme conditions such as high temperature, high strain rate and fatigue testing. The second part will deal with the application of nanomechanical testing to investigate consequences induced by manufacturing processes. More specifically, various cases ranging from severe shot peening to sliding friction contacts are investigated. The last part of this presentation will focus on a new high-temperature nanoindentation procedure, the High Temperature Scanning Indentation (HTSI) method [2], developed on purpose to investigate the thermal stability of these surface-processed materials.

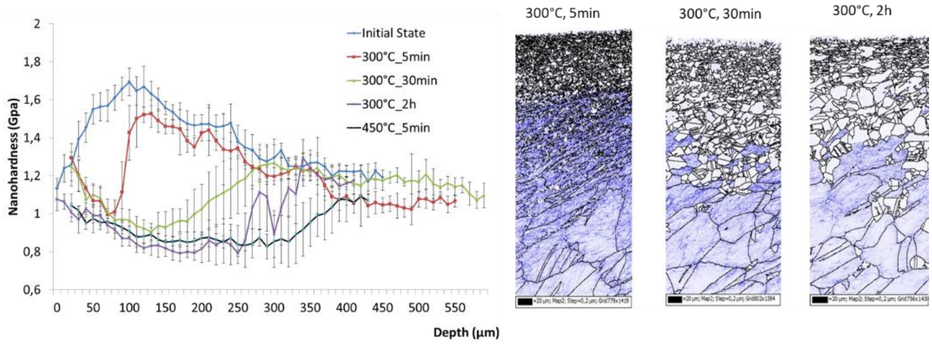

Fig. 1: investigation of thermal stability of a Tribologically Transformed Surface resulting of high speed friction on a Cu bar using nanoindentation testing (left) and EBSD (right)

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The comparison of micromechanical properties of the compositionally complex transition metal nitride coatings deposited by different reactive sputtering techniques

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Keywords: transition metal nitrides, reactive sputtering, DC magnetron sputtering, HiTUS, nanoindentation

The multicomponent transition metal (TM = Ti, Nb, V, Ta, Zr, Hf**)**-based nitride ceramics belonging into an extended family of compositionally complex ceramics with excellent mechanical properties which make them candidates for the extreme condition applications. The aim of the current work was to compare micromechanical properties of the multicomponent TiNbVTaZrHf–xN (x = nitrogen flow in sccm) coatings deposited by the reactive DC magnetron sputtering (DCMS) and reactive High Target Utilization Sputtering (HiTUS).

DCMS deposited TiNbVTaZrHf metallic coatings exhibited textured bcc structure; despite significant local variations of the concentrations of individual TM coatings, they formed solid solution attributed to high entropy alloys. Analogous coatings in HiTUS were amorphous. Structure and composition of TiNbVTaZrHf–xN coatings were practically identical both in DCMS and HiTUS and depended on the nitrogen flow, x. At low x, nitrogen concentrations were almost linearly proportional to x. At and above critical flow, x_c, nitrogen saturation and stoichiometry around 1.0 were achieved. Coatings exhibited textured fcc structure Despite local deviations of the concentrations TM and nitrogen from the ideal theoretical concentrations, the structure of all coatings still corresponded to the single phase solid solutions analogous to the high entropy stabilized ceramics.

Nanoindentation measurements of hardness and indentation moduli revealed their close correlations with nitrogen stoichiometry. DCMS-made coating exhibited hardness ~ 40 GPa while that in HiTUS coatings was ~ 33 GPa. The corresponding indentation moduli were ~ 490 GPa vs ~ 400 GPa. They were achieved at optimized x resulting in nitrogen stoichiometry of 48 at % and 45 at%, respectively. However, the ratio H_{IT}/E_{IT} and limited number pillar split tests indicated very low ductility and fracture toughness only in the range of 1 MPa.m$^{1/2}$.

The work confirmed that both reactive DCMS and reactive HiTUS can produce compositionally complex transition metal-nitride ceramics with different stoichiometry which controls their mechanical properties.

Acknowledgement

This work was supported by the Slovak Research and Development Agency [projects APVV APVV-21-0042], Research Agency of the Ministry of Education, Science, Research and Sport of the Slovak Republic [project VEGA 2/0083/23] as well as by the International Visegrad Fund [project JP39421, V4-Japan Joint Research Program].

Plastic instabilities and strain recovery in amorphous LiPON thin layer

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Keywords: Solid state batteries, Li-ion batteries, nanoindentation, plastic instability, strain recovery.

Bulk materials typically deform uniformly, but when their size decreases significantly, deformation becomes uneven and unpredictable. New probabilistic methods were needed to the in depth understand of random local plastic events, with the authors contributing through a custom-developed in situ nanoindenter [1]. In the presentation, the focus is on a novel-like microdeformation phenomena in amorphous materials.

Over the past decades, the demand for portable power has risen, leading to research on solid-state batteries (SSB) as safer alternatives to lithium-ion batteries. Lithium phosphorous oxynitride (LiPON) is a crucial electrolyte for SSBs due to its sufficient ionic conductivity. Understanding the mechanical behavior of LiPON films is crucial for technology development.

Previous studies [2] noted unexpected ductility and strain recovery in amorphous LiPON, with sharp-ended tip indentations revealing pile-up formation and densification as the main deformation mechanisms. Our work presents nanoindentation experiments with a spherical tip, revealing a novel mechanical behavior—a sudden deformation event followed by slow strain recovery during unloading. This unique deformation is likely linked to the material's special structure, featuring isolated phosphate tetrahedra embedded in a Li matrix with occasional N bridge bonds between tetrahedra. In the presentation, the author will report on over a hundred indentations, examining how instability depends on strain rate and the sharpness of the indenter's tip.

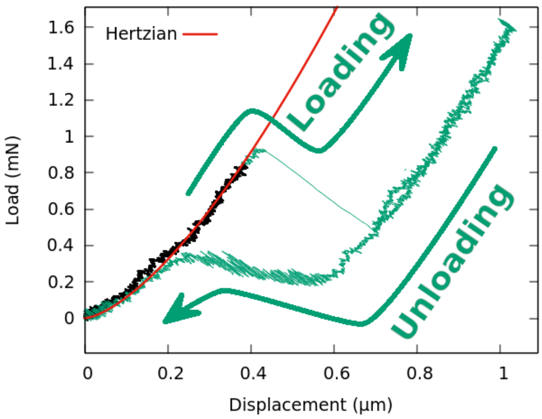

Fig. 1: A representative indentation curve on LiPON with a spherical-shaped indenter tip

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Surface functionalization in selective laser melted 17-4 PH by plasma polishing and interstitial diffusion hardening

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Keywords: selective laser melting, 17-4PH, plasma polishing, interstitial diffusion hardening

Developments in processing technology and feedstocks are key drivers for new product innovations in the field of additive manufacturing. In the area of complex and filigree geometries, additive manufacturing technologies are often superior to conventional processes. Selective laser melting (SLM) as powder bed process allows components of different scales to be manufactured by variation of the grain size range of the feedstocks. However, the surface quality achieved is a critical factor. The powders used as feedstock fundamentally limit the surface quality of SLM components. Particle contamination on the surfaces of the parts can remain rounded or agglomerated contributing to a very rough surface at the microscale. Furthermore, the manufacturing advantages of a closed component design lead to limitations in the mechanical finishing process, especially regarding undercuts and cavities. In contrast, surface quality has gain an increasing role. In addition to corrosion protection requirements, demands for wear resistance get more and more frequent. This study deals with the development of a process chain for the surface functionalization of selective laser melted 17-4 PH by plasma polishing and interstitial diffusion hardening. In this context, both the leveling of the surface topography and the development of graded coating properties are of particular interest. While plasma polishing offers appropriate potential for the formation of smooth surfaces, low-temperature diffusion processes can increase wear resistance while avoiding component distortion as a result of heat treatment. Both individual technologies each promise high application potential, which can be exploited to the optimum through the development of a process chain.

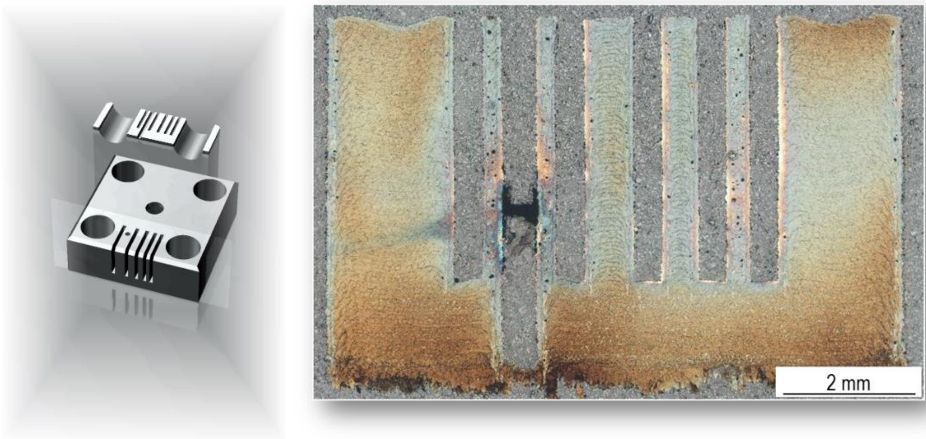

Fig. 1: 17-4PH SLM sample (right) and Beraha II etched cross-section view (left) of the print direction.

Acknowledgement

This work was co-financed with tax revenues based on the budget approved by the members of the Saxon State Parliament and a grant of the Romanian National Authority for Scientific Research and Innovation, CCCDI – UEFISCDI, project number COFUND-M-ERANET-3-HardCoat-1, no. 311/2022 (INOE), within PNCDI III and partner project M-ERA.NET Technology Agency of the Czech Republic (No. TA ČR TH80020005)

SACHSEN

Contact damage of multi-material laminar ceramics: effect of layers architecture and residual stress

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Keywords: laminar ceramics, contact damage, nanoindentation, crack resistance

Multimaterial laminar ceramics offer high mechanical strength and high tunability in terms of resistance to fracture and extension of cracks. In the present study, we investigated the damage response of multi-material laminar ceramics produced by stacking alumina and $MgAl_2O_4$ layers to Vickers indentation and Hertzian contact. Laminar ceramics with a $MgAl_2O_4$/alumina/$MgAl_2O_4$ and alumina/$MgAl_2O_4$/alumina architecture were produced through spark plasma sintering of layers shaped by tape-casting. Hertzian indentation was performed using a tungsten carbide sphere; the results showed that compressive stress produced in $MgAl_2O_4$/alumina/$MgAl_2O_4$ resulted in a significant increase, ca. 70%, of the strength of the surface layer of multi-material structure compared to bulk $MgAl_2O_4$ ceramics; in contrast, alumina/$MgAl_2O_4$/alumina architecture exhibited a lower resistance compared to the bulk alumina sample. Also, the crack resistance (CR) corresponding to the load leading to an average of two radial/median cracks per indent was measured by performing Vickers indentation at different loads. . The results revealed that the $MgAl_2O_4$/alumina/$MgAl_2O_4$ structure is characterized by a higher damage resistance, which corroborates the Hertzian indentation results. The distribution of residual stresses within the layers was investigated using nanoindentation. The correlations between the residual stress distribution and the overall performance of laminar samples is critically discussed.

Acknowledgement

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Micromechanical testing of irradiated concrete

Fracture properties of Calcium-Silicate-Hydrates exposed to γ-irradiation and different relative humidities

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Keywords: cement paste, focused ion beam, γ-irradiation, tensile strength, fracture energy

Concrete and its main binding phase, the cement paste, deteriorate due to neutron and gamma radiation when exposed at nuclear power plants [1]. These processes lead to a decrease in mechanical properties, a phenomenon documented on a macro-scale [2]. However, the underlying mechanisms responsible for concrete degradation originate from the nano to micro-scale. Gamma radiation causes water radiolysis and gamma heating, predominantly causing the drying of Calcium-Silicate-Hydrates, the main micro-scale component of cement paste [1]. As drying competes with other mechanisms such as carbonation and hydration, testing of gamma-irradiated samples under specific humidity conditions become necessary [3]. To assess local fracture properties at the micro-scale, specimens with a specific geometry, prepared using focused ion beam technology, were prepared [4]. Samples of cement paste made of CEM I-42.5R and irradiated using 200 TBq ^{60}Co source up to dose of ~13 MGy were tested. During the irradiation samples were stored in three levels of relative humidity conditions [3]. Micro-cantilevers with a triangular cross-section were prepared on each sample in two selected phases using a dual-beam FIB-SEM microscope, Tescan Lyra3 GMU. A Nanoindenter Hysitron TI-980 (Bruker Company) was used to obtain tensile strength, fracture energy, and Young's modulus for irradiated and non-irradiated samples.

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Micromechanical characterization of γ-irradiated cement paste exposed to different relative humidity conditions

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Keywords: cement paste, γ-irradiation, nanoindentation, water radiolysis, drying

Concrete in nuclear power plants used as biological shielding around the active zone is exposed to radiation and thermal strains [1]. The decrease in mechanical properties, primarily the compressive strength of concrete, was previously reported in [2-3]. The deterioration is mainly caused by neutron radiation, gamma ray radiation, drying, and heating [1]. Gamma rays primarily affect the water in Calcium-Silicate-Hydrates due to water radiolysis, causing concrete drying [4]. It was already reported that the relative humidity level in the environment significantly affects the micromechanical properties of 28-day-old cement paste exposed to gamma radiation (absorbed dose 2.88 MGy) [5]. In this study, 20 months of well-hydrated cement pastes samples from Portland cement CEM I-42.5R (Českomoravský cement, Czech Republic) were gamma irradiated by 200 TBq by ^{60}Co source with the total absorbed dose of ~13 MGy. During the irradiation, the samples were stored at specific relative humidity conditions of 11%, 33%, 76%, 96%, and water. Grid nanoindentation was performed on polished sample surfaces to access micromechanical properties e.g., Young's modulus and hardness of irradiated and control samples. The results were supported by other techniques, such as X-ray diffraction and mercury intrusion porosimetry, to decouple the main mechanisms: drying, carbonation, and water radiolysis.

Acknowledgement

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Optimization of FIB milling procedure for micromechanical testing of cement pastes

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Keywords: cement paste, focused ion beam, γ-irradiation, nanoindentation

Cement paste is a very heterogeneous non-conductive material, with minor crystalline and dominant amorphous phases. It is composed of several main hydration products, namely Calcium-Silica-Hydrates, Portlandite, residual clinker, porosity, and other minor phases such as ettringite and monosulfate [1]. These phases exhibit variable chemical composition (e.g. density or Ca/Si ratio) and different mechanical properties. To assess micro-mechanical properties of the micrometer-sized phases, nanoindentation can be employed as the primary tool to a certain extent [2]. Accessing fracture properties requires fabrication of small micro-sized specimens with various geometries. The task can be performed using focused ion beam (FIB) technology [3] with finely tuned parameters. Cement paste is susceptible to the heat generated during FIB milling, causing irreversible phase changes [4].

This study focuses on optimizing of FIB preparation process for micro-cantilevers to avoid microstructural phase changes in a reasonable time frame. The sample used in this study was prepared from Portland cement CEM I-42.5R (Českomoravský cement, Czech Republic). Cantilevers with a triangular cross-section [3] were prepared in four selected phases: inner and outer products, Portlandite, and clinker [1], using a dual-beam FIB-SEM microscope, Tescan Lyra3 GMU. The effect of specific fabrication procedure and milling parameters, such as accelerating voltage, current, and time on geometrical precision, sample morphology and resulting micromechanical parameters were evaluated and optimized.

Acknowledgement

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Nanomechanical properties of neutron-irradiated concrete

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Keywords: concrete, nanoindentation, neutron irradiation, mechanical properties

Concrete exposed to reactor irradiation undergoes atomic-level degradation due to the influence of neutrons, secondary gamma rays, and associated gamma heating [1]. The radiation-induced damage decreases the mechanical properties of the concrete and affects its performance as a construction material [2-4]. The concrete samples analyzed in this work were prepared from blended Portland cement CEM II 32.5R mixed with siliceous aggregates with a size of 0 – 2.5 mm. The material underwent irradiation in the LVR-15 research reactor in Research Centre Řež to the target neutron fluence of 1.6-1.8×10^{19} n/cm^2 (E>0,1 MeV) and received the gamma dose of ~ 500 MGy. The temperatures during irradiation were kept within a range of 50 – 60 °C. The mechanical properties of the individual phases in the cementitious matrix and in the aggregate after neutron irradiation were assessed by the nanoindentation. In order to track the evolution of the nanomechanical properties of the individual components under radiation, a comparison of mechanical properties between the material after neutron irradiation, gamma irradiated concrete (13 MGy), and pristine concrete was made.

Acknowledgement

This work was financially supported by the project of the Czech Science Foundation grant number 23-05435S. The presented results were obtained using the CICRR infrastructure, which is financially supported by the Ministry of Education, Youth and Sports - project LM2023041.

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Experimental challenges and coupled methods

High strain rate persistence of the strength anomaly in $L1_2$ intermetallic compound Ni_3Si evidenced by nanoindentation at elevated temperatures

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Keywords: nanoindentation, high strain rate, high temperature, strength anomaly

$L1_2$ intermetallic compounds are essential constituents of the superalloys used in jet engine turbines. They derive their exceptional mechanical properties at high temperatures from the so-called strength anomaly, which has received wide attention as early as in the 1970s. However, most investigations to date have been performed at moderate strain rates and it is still unclear whether the anomalous behavior endures at high velocities, e.g. during a collision such as a bird strike. Novel experimental methods are required.

Nanoindentation is a versatile method for measuring the mechanical properties of materials at the nanoscale, which is frequently carried out at elevated temperatures. However, it has been so far limited to slow strain rates < 0.1 s^{-1}. The presentation will highlight recent developments in the field, which have extended the permissible strain rate range to 100 s^{-1} at room temperature [1]. Finally, both "extreme" capabilities are combined in a setup developed at Texas A&M University.

Using this setup, the influence of high strain rates on the strength anomaly of $L1_2$ intermetallic compounds is investigated by nanoindentations on Ni_3Si under varied temperatures and strain rates. It is concluded that the anomalous range is extended under high strain rate conditions.

Acknowledgement

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Experimental Progress in High Constant Strain Rate Nanoindentation

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Keywords: nanoindentation, high strain rate, experimental

Knowledge about the local mechanical behavior at high deformation rates is crucial to increase the safety of components that involve impact loading such as bird impacts in the aviation industry, smartphone displays, automobile crashes and many others.

Nanoindentation has been used extensively in the past decades to study the local deformation behavior of various types of materials but these were primarily limited to low strain rates: 10^{-3} – 10^{-1} s^{-1}. To be able to study deformation behavior at high strain rates, there is a need to not only use fast signal processing components but to also develop experimental methods and analysis techniques for high speed nanoindentation tests.

This talk will focus on both technical implementations and further development of previously established methods [1] to analyze high constant strain rate nanoindentation data. A displacement-controlled instrument will be presented, which was designed around piezoelectric transducers and fast electronics allowing sampling rates of up to 1 MHz. We will show experimental measurements at strain rates of up to 10^4 s^{-1} and discuss current bottlenecks, both in terms of resonance effects and time constant corrections.

Acknowledgement

This project has received funding from the European Research Council (ERC) under the European Union’s Horizon 2020 research and innovation programme (grant agreement No 949626)

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Fatigue testing of coatings by using the dynamic capabilities of a nanoindenter

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Keywords: fatigue testing, nanoindentation, spherical indenter, thin films

The fatigue behavior of thin coatings is not well known due to a lack of test methods for this property. However, in many applications they are exposed to cyclic loading that can result in catastrophic failure of the whole component. Therefore, a characterization of the coating material only by hardness and modulus may be insufficient. Impact testing [1] is one technique that tries to get access to the fatigue behavior. However, the test conditions agree more to that of impinging particles that collide with the surface with high speed and do not reproduce the conditions of a cyclic loading during a permanent static contact of the counterpart with the surface.

The ZHN nanoindenter has the ability to produce large oscillations additional to the static force with force amplitudes up to 100mN or more in a frequency range between about 5 Hz and 50 Hz. By use of a spherical indenter of a radius between 5 µm and 10 µm it is possible to adjust the position of the maximum von Mises stress into the coating or close to the interface for coatings with a thickness of 5µm or less. This allows the execution of fatigue test that can represent the conditions in an application. The results of such fatigue tests are presented for coatings of different materials and thicknesses (see fig 1 as example). It is shown that characteristic results can only be obtained when the force amplitude comes close to the applied static force. By this test method it is possible to distinguish between good and bad fatigue behavior for cycle numbers of several ten thousand.

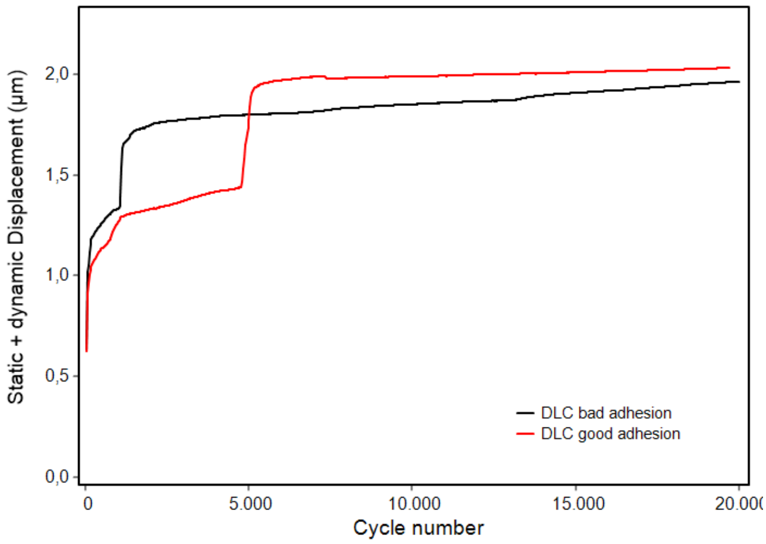

Fig 1: Example for two fatigue test results of DLC coatings

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Nanoindentation methods for analysis of thermally activated processes at elevated temperatures

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Keywords: nanoindentation, high temperature, ultrafine-grained microstructure, strain rate sensitivity

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

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

The temperature matching procedure in particular can be time-consuming, which limits the applicability of conventional high temperature nanoindentation to materials with sufficiently stable microstructures at the testing temperature. To overcome some of these limitations, the High Temperature Scanning Indentation (HTSI) technique was recently presented by Tiphene et al. [1]. It utilizes a large amount of high speed tests (on the order of 1s each) to generate a high density of data points and measure mechanical properties during thermal ramping of a sample. This allows the investigation of dynamic processes like recrystallization.

In this contribution, the HTSI technique is applied to investigate the thermal stability of ultrafine-grained CuSn5 and CuZn5 alloys processed via high pressure torsion [2,3]. Samples are subjected to different thermal cycles and hardness and strain rate sensitivity are evaluated as a function of temperature. Microstructural analysis via SEM and additional DSC measurements are used to validate the experimental findings.

Acknowledgement

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High Strain Rate Nanoindentation – Recent Advances and Perspectives

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Keywords: In-situ, High Temperature, Low Temperature, Micromechanical Testing, High Strain Rate

The strain rate dependence of mechanical properties of materials at high strain rates, is of great interest for many applications including metal forming, machining, crashworthiness, projectile impact, etc. It therefore is desirable to extend high strain rate capabilities of nanoindentation to strain rates beyond the quasi-static regime, taking advantage of relatively simple sample preparation, high-throughput testing capabilities and comparably small equipment.

This talk will focus on recent developments in instrumentation and testing protocols for high strain rate testing from 0.0001/s up to 10'000/s with high-speed actuation and sensing capabilities. Moreover, we will elaborate on high strain rate testing performed at high and low temperatures as well as under variable humidity, and how the resulting multi-dimensional data is useful for understanding fundamental material behaviour and advancing engineering objectives. The challenges of such experiments and the associated technological and test protocol advances will be discussed along with select case studies.

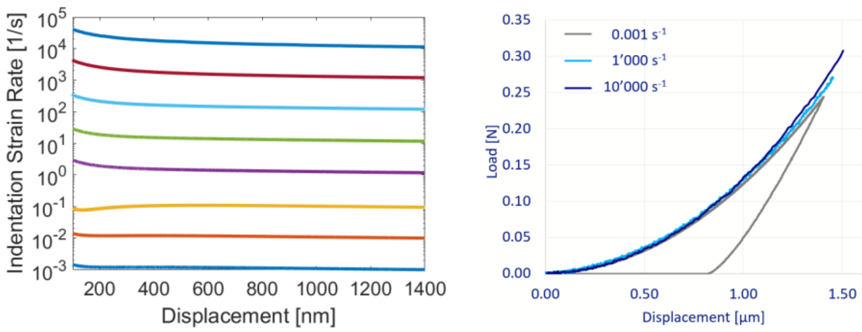

Fig. 1: Constant strain rate nanoindentations on fused silica. Strain rate vs depth and load vs displacement curves.

Unraveling acoustic emission by coupling it to micromechanics

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Keywords: micromechanics, micropillars, acoustic emission, strain bursts, machine learning

Deformation of most materials proceed in a sequence of sudden local deformation events. Acoustic emission (AE) measurement is a non-destructive and widely used method to monitor such processes. If the magnitude of the local events is large, the released elastic energy induces surface vibrations that can be detected. The measured signal is characteristic of the deformation event, however, due to the complex mechanisms involved and the difficulty of observing the deformation event itself makes interpretation of AE data a very challenging task [1].

In this talk we show results on coupling AE with micromechanics in order to obtain a more detailed understanding of the AE signals. Micropillars were milled from a Zn single crystal and oriented for basal slip and then mounted on an AE sensor and compressed in situ in a scanning electron microscope using a device specially designed for this purpose [2]. We found that the acoustic events detected during compression are in perfect correlation with the stress drops measured by the micromechanical device and the statistical analysis revealed complex spatio-temporal dynamics of dislocations [3]. In the second part of the talk we investigate what information is carried by AE signals about the local events. To this end, we apply machine learning of the AE dataset and after proper training a convolutional neural network using wavelet transforms of the AE signals we were able to predict the force-time curves of individual micropillars to great precision (Fig. 1). Our results open new perspectives in understanding the information content of AE signals and to interpret them.

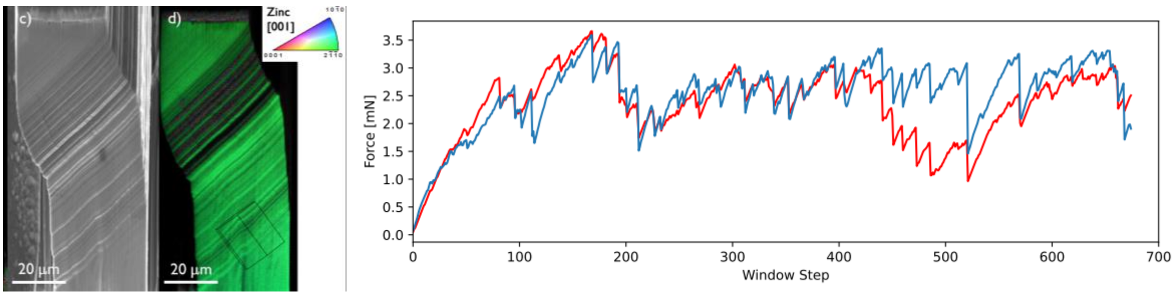

Figure 1: A compressed Zn micropillar and EBSD measurement performed on it (left panel) and the measured force-time curve (blue) and the one predicted with machine learning purely from the measured AE data (red, right panel).

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High-resolution measurement of strain by tracking of nanoparticles

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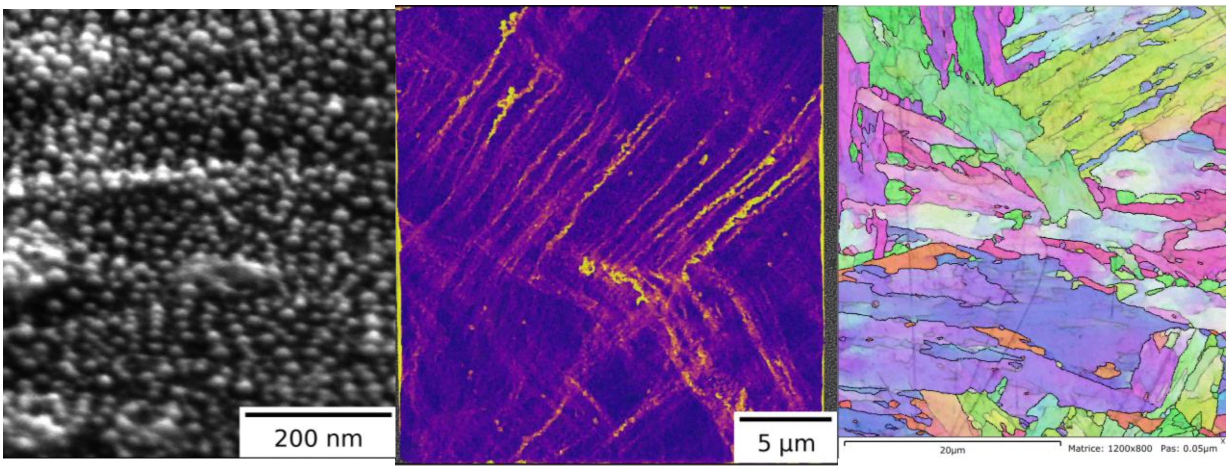
Keywords: strain measurement, pulsed-laser induced dewetting markers, tempered martensitic steel, tracking algorithm, slip bands statistics

Measuring the strain with a high resolution allows to better understand the plasticity, embrittlement mechanisms and ruptures of materials. That is why we developed a new method to measure strain fields with high-resolution scanning electron microscope (SEM) images. It offers a resolution of 30 nm and a precision of 1 nm.

A pulsed-laser induced dewetting of gold nanofilm creates a high density of small nanoparticles (~17 nm) (Fig1-a) [1]. It is possible to distinguish the markers one from each other and so to track them during the deformation of the material. The relative displacement of the markers allows us to measure the deformation on the surface during a tensile test. The tracking algorithm is really fast (a few seconds on a standard computer) and offers a lot of possibilities: for instance the stitching of several images to get a larger field of view or also the correction of the SEM aberrations that appears at high magnification.

The method was successfully applied on the tempered martensitic steel Eurofer97 (E97)(Fig1-b) and gives access to the statistic of the slip bands (width and strain) depending on the orientation of the grains. The propagation of a slip band on the other grains is also studied. EBSD map taken before the formation of gold nanoparticles on the surface helps to understand the deformation observed(Fig1c).



(a) (b) (c)

Fig. 1: (a) Tilted view of gold nanoparticles taken with the SEM. (b) Von Mises deformation of E97 obtained by tracking of nanoparticles. (c) An electron backscattered diffraction map of E97 which allows to better understand the deformation.

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Microeletronic materials

On the phase-engineered novel phase of Silicon

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Keywords: Phase transformations, Nanoindentation, Mechanical properties, nanoECR, Solar cells, Rhombohedral phase of silicon

Among the thirteen polymorphic phases of Si, the ambient temperature stable body-centered cubic (bc8) and rhombohedral (r8) polymorphs have gained significant interest due to their attractive optical and electronic properties suitable for photovoltaic applications. Recent first-principles calculations have shown that the r8 phase of Silicon (r8-Si) is a narrow bandgap semiconductor and hosts a quasiparticle spectrum. Due to its high dielectric function, it shows exciting optical properties such as a high absorption coefficient (in 1 - 4 eV energy range of the solar spectrum) compared to the other forms of Si.[1] The boosted light absorption translates into an improvement of current in the optoelectronic devices. Our In situ high temperature micro-Raman studies along with high-resolution cross-sectional TEM analysis reveals that the overall transformation observed is directly from r8→polycrystalline dc-Si and bc8→polycrystalline dc-Si rather than through other metastable phases such as Si-XIII/hd-Si with increasing the annealing temperature from 25°C to 220°C. Our systematic in situ study high temperature microRaman study provides evidence for a few earlier predictions and clarifies ambiguities involved in understanding the annealing behavior and transformation pathways of two high-pressure phases of Si at elevated temperatures.[2]

The rhombohedral phase of silicon is fabricated by nanoindentation over a large-area within the diamond cubic Si wafer using spherical nanoindentation. The ultraviolet and visible spectrum showed a ~50% lower reflectance for the rhombohedral Si compared to the pristine Si. The estimated refractive index of ~6.7 at 630 nm for the rhombohedral Si is ~35% higher than the diamond cubic Si, which agrees well with the theoretical calculations. The rhombohedral Si with enhanced light absorption ability is utilized as a solar absorber layer for the bare p-n junction Si solar cell and observed to show ~10 times improvement in the photocurrent density.[3]

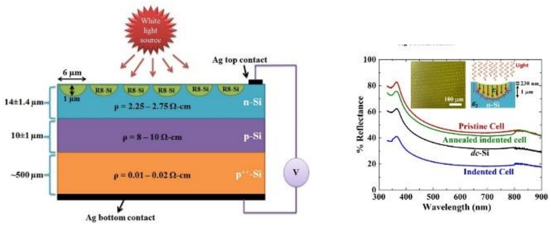

Fig. 1: Employing r8 phase of silicon for absorbing more solar radiation.

Acknowledgement

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3D Tomography on Advanced Photovoltaic (PV) Structures – Examples of Good Practice

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Keywords: Scalpel AFM, removal rate, microcrystalline silicon, electrical conductivity.

The use of doped microcrystalline silicon (µc-Si:H) thin films as carrier selective contacts in the back side of silicon heterojunction (SHJ) solar cells, allows this technology to achieve high world record conversion efficiencies (> 26%).[1] A study of surface-dependent growth of such thin films is crucial to understand the phenomenon of 'interband tunneling' of carriers, present in tunnel interdigitated back-contacted technology (tunnel-IBC).[2] To study the structural and electrical properties of such films, through the evolution of electrical resistances on the nanoscale, 3D tomography using C-AFM in contact mode (also called scalpel AFM) can be a promising technique.[3] With this technique, the sample material can be removed in thin slices, layer by layer, and the conductivity can be measured simultaneously.

In this work, we report an approach for a reliable scalpel AFM, based on our experiences through a series of measurements. Finding a low and stable removal rate during each scan, using either a full boron-doped diamond probe or a probe with proficient doped diamond coating, is one of the main objectives of such an experiment. Scan parameters like normal force applied to the tip, scan rate, the number of points/line, direction of the scanning, etc. can alter the removal rate significantly. It is found that proper cleaning of a contaminated tip is advantageous for maintaining good process consistency. Optimizing all the above-described procedures, it's possible to generate a definitive 3D tomogram.

In addition to the results achieved on microcrystalline silicon thin films, we will represent a few results from our previous work on hole-selective passivating contacts, consisting of an interfacial layer of silicon oxide (SiOx) and a layer of boron-doped SiCx(p) using tomographic AFM.[4]

Acknowledgment

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

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Keywords: BGA interconnections, Polymer Core Solder Ball, micromechanics, multi-scale analysis

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) (Figure1 a,b). 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 (Figure1 c), the characterization of the mechanical properties (Young modulus (E) and Yielding strength (σ)) 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 (Figure1 d), 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].

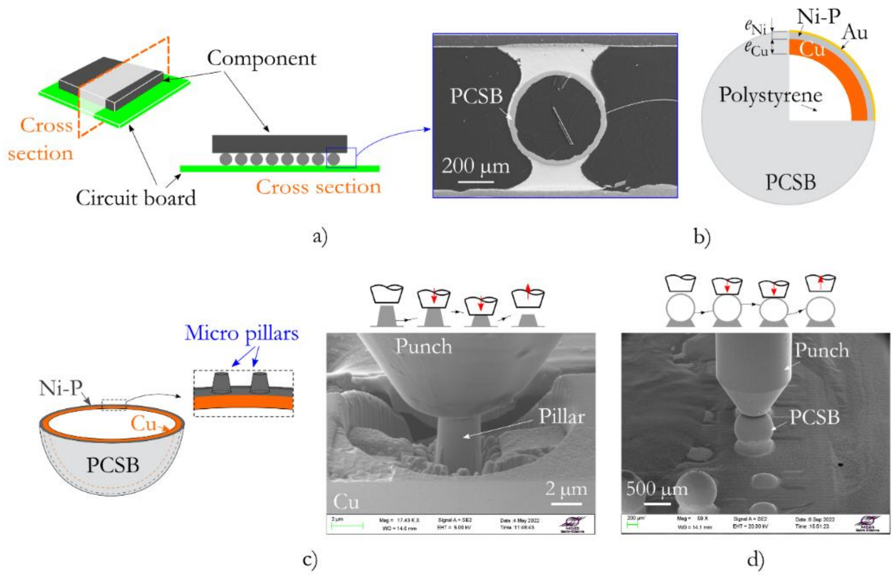

Fig. 1: a) BGA interconnection with PCSB, b) detail of the coatings of the solder ball, c) micro pillar compression tests (microscopical scale) and d) PCSB compression tests (mesoscopical scale).

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Micromechanical testing of polymers

Comparison of Spherical Indentation Analysis for Soft Polymers in Bio Applications

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Keywords: soft polymers, spherical indentation, Hertzian elastic contact, elastic-viscoelastic correspondence, load relaxation data

Interest in mechanical characterization of soft polymers in bio applications is growing rapidly, due to theirs use in tissue engineering. For example, hydrogels for 3D bioprinting must feature tunable stiffness to enhance its biocompatibility so cells can spread, proliferate, and allow to receive nutrients and remove waste via hydrogel porosity.

Depth sensing indentation records full indentation load-depth-time (P-h-t) profile which can be analysis for material mechanical properties. Three different analyses were compared by means of feasibility and applicability to spherical indentation data. Single indentation data set can be deconvoluted for (i) elasticity from P-h curve of loading segment (ii) viscoelastic behavior from P-t load-relaxation segment, and (iii) elastic-plastic properties upon P-h unloading segment.

In the load-relaxation model, an exponential decay equation is fit to a displacement hold segment to extrapolate the load after infinite time. This model has proven the high applicability to soft polymers used for bio applications.

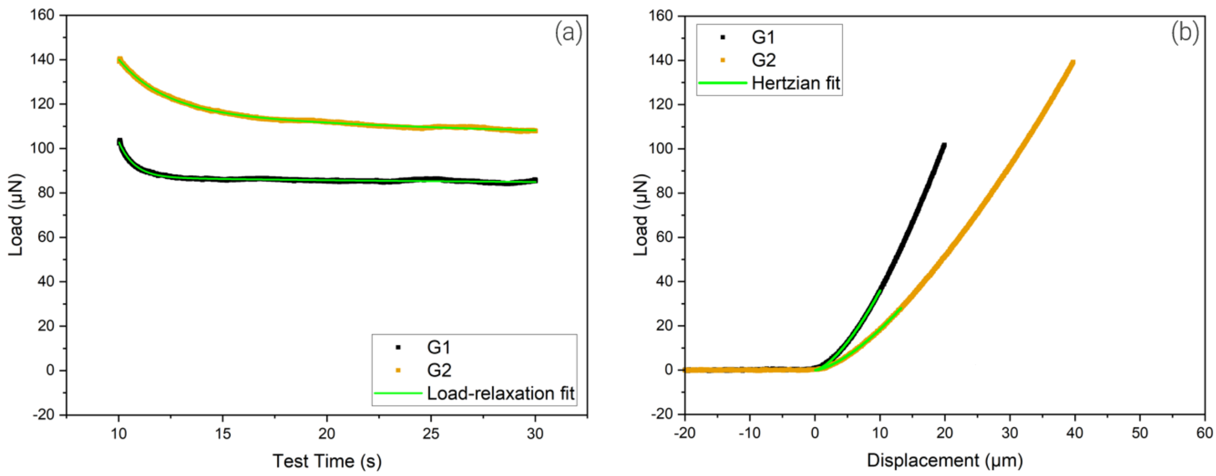

Fig. 1: Analysis of spherical indentation data: (a) viscoelastic load-relaxation model (b) Hertzian elastic contact model.

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Macro-, micro- and nanomechanical characterization of crosslinked polymers with very broad range of mechanical properties

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Keywords: microindentation, nanoindentation, crosslinked amorphous polymers, correlations

We compared of macro-, micro- and nanomechanical properties of a series of eleven highly homogeneous and chemically very similar polymer networks [1]. By means of synthesis parameters, the mechanical properties of the samples were deliberately changed from very hard and stiff (elastic moduli 4 GPa), through semi-hard and ductile, to very soft and elastic (elastic moduli 0.006 GPa).

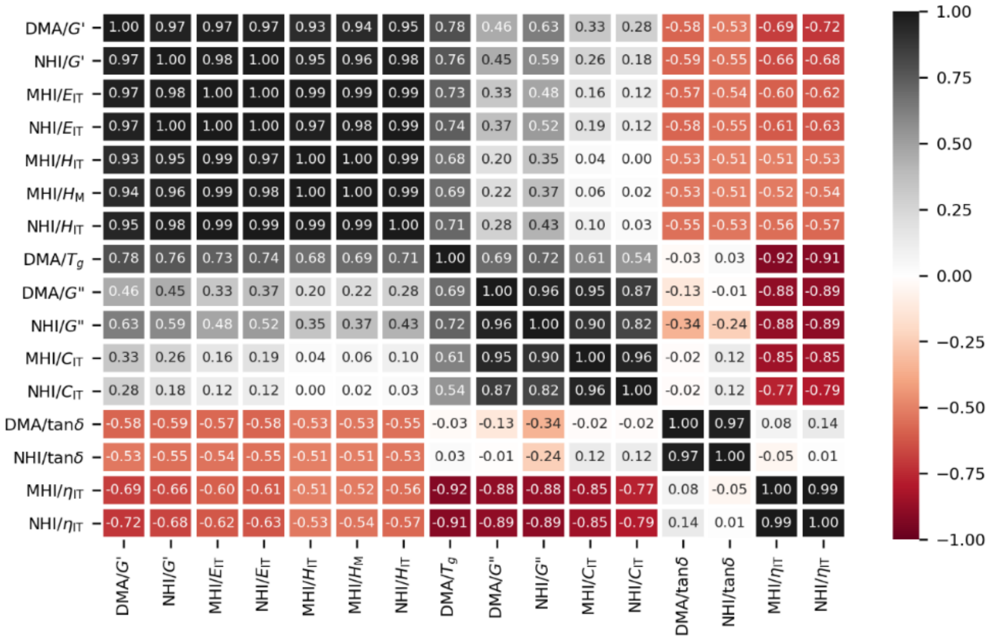

Figure 1: Correlation matrix table showing Pearson's coefficients *r* for all pairs of experimentally determined properties in macro-, micro- and nanoscale: storage and loss modulus, damping factor and glass transition temperature from dynamic mechanical analysis (DMA/G', DMA/G'', DMA/tan(δ) and DMA/T_g), analogous properties from dynamic nanoindentation experiments (NHI), indentation hardness, modulus, creep and elasticity from quasi-static microindentation experiments (MHI/H_{IT}, MHI/E_{IT}, MHI/η_{IT} and MHI/C_{IT}), and analogous properties from quasi-static nanoindentation (NHI).

Mechanical properties at all length scales showed similar trends and interesting correlations as shown in (Fig. 1). This contribution will summarize the key conclusions from our detailed study [1].

Acknowledgement

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Comparison of macroscale, microscale and nanoscale creep behavior of UHMWPE and PEEK polymers used in total joint replacements

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Keywords: macroscale, microscale and nanoscale creep, polymers, total joint replacements

We compare macroscale tensile creep (macrocreep), microscale indentation creep (microcreep), and nanoscale indentation creep (nanocreep) of three synthetic polymers: virgin ultrahigh molecular weight polyethylene (UHMWPE), highly-crosslinked UHMWPE (UHMWPE-XL) and polyether-ether ketone (PEEK). These polymers are employed as liners in total joint replacements (TJR) in orthopedic surgery. Their mechanical performance is one of the factors influencing the TJR lifespan [1].

The prediction of macrocreep behavior from micro- or nanoindentation experiments is useful, because the macrocreep experiments are both material-consuming (at least 3 specimens ca 10 cm long) and time-consuming (from hours to weeks), while microcreep experiments can be carried out with a small sample (dimensions in millimeters) and much shorter times (from tens of seconds to minutes). Our previous work comparing macro- and microcreep of three common commercial polymers with a broad range of properties (polyethylene, polypropylene and polystyrene) proved that the macroscale and microscale creep properties correlated quite well [2]. This study extends our previous work in two aspects: (i) we compare polymers with more similar properties and (ii) we correlate the macroscale tensile creep not only with microindentation, but also with nanoindentation creep.

The results were evaluated with our own freeware Python package MCREEP [2, 3]. The package can fit several models to both tensile and indentation creep data in a well-defined, consistent, and user-friendly way. This enables us not only to describe the creep behavior, but also to compare the performance of models at all length scales, and to predict the long-term creep properties.

The microindentation experiments confirmed our expectation that the microcreep performance of UHMWPE and UHMWPE-XL is almost identical, whereas PEEK polymer exhibited significantly higher creep resistance. Moreover, we have re-demonstrated that the simple empirical two-parameter *power law model* yields better predictions of long-term creep behavior than the more sophisticated multiparameter *elasto-visco-plastic models* recommended by Oyen [4] or Mencik [5]. Finally, we compared other micromechanical properties, such as indentation modulus and hardness of all three polymers. The macrocreep and nanocreep experiments are ongoing.

Acknowledgement

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Comparison of Berkovich and spherical tip indentation for determining the Young's modulus of polymer thin films encapsulated by a dielectric capping

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Keywords: nanoindentation, Berkovich tip, spherical tip, thin films, polymer, acrylic resin.

Pixel integrated micro-lenses for CMOS image sensors consist of a stack of polymer acrylic resin films encapsulated by a dielectric layer (fig. 1, a). Due to the mismatch of thermomechanical properties, adhesive or cohesive ruptures can occur, leading to reliability issues requiring the knowledge of the polymer thermomechanical properties. Nanoindentation is a standard method for determining Young's modulus of thin films. However, when performing temperature-dependent nanoindentation studies directly on the aforementioned polymer film, contamination of the tip can occur near or above the glass transition temperature leading to estimation error at the end. Therefore, evaluating the complete lens stack is the only solution. The standard Berkovich tip creates cracks in the dielectric (fig. 1, b), leading to potential tip contamination. As a first step, this work, focuses on room-temperature experiments to evaluate the use of a spherical tip (radius: 5 and 50 µm), less likely to create cracks, for the direct determination of the Young's modulus of the encapsulated film. It is determined using a multilayer model [1, 2], the limits of which are investigated based on finite element simulations. To validate the proposed method, different configurations are evaluated. First, a reference value of the Young's modulus of a 3 µm-thick resin layer is determined without dielectric capping. Second, the effect of dielectric thickness on Young's modulus value is studied. Similar Young's modulus values are obtained for all samples and indentation tips (fig. 1, c). Moreover, the use of a 50 µm radius tip, prevents the generation of cracks and therefore the potential contamination of the tip. These trials validate the methodology and its use for future temperature-dependent nanoindentation studies.

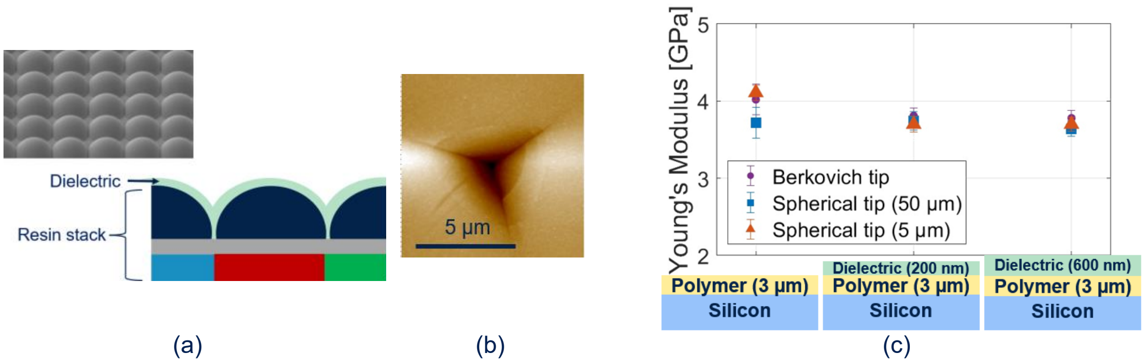

Fig. 1: (a) Microlenses array schematic. (b) AFM image of indentation cracks. (c) Extracted polymer Young's modulus.

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Challenging materials

The effect of crystal anisotropy on fracture toughness and strength of ZrB_2 microcantilevers

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Keywords: crystal anisotropy, fracture toughness, strength, slip systems, microcantilever bending, ZrB_2

The influence of crystal anisotropy on the micromechanical properties of ceramic grains plays an important role in the design of the macromechanical performance of bulk polycrystalline samples. To this end, the effect of crystal orientation on fracture toughness and strength was investigated by microcantilever bending experiments combined with Finite Element Method (FEM) simulations in grains of a polycrystalline ZrB_2 sample. The sample was prepared by hot pressing and the crystal orientations were determined by electron backscatter diffraction after careful surface preparation. The bending tests were carried out on notched and unnotched microcantilevers cut from specific grains along the prismatic (⊥ to c-axis), basal (∥ to c-axis) and intermediate (45° to c-axis) directions of ZrB_2 crystals using focused ion beam milling. The fracture toughness and strength were determined by FEM-derived analytical solutions. The fracture strength was similar with values of about 11-12 GPa in every direction. Enhanced plasticity was found in the intermediate direction of unnotched beams as compared to the other two brittle orientations (Fig. 1). The fracture toughness was found to be 30% higher in the intermediate direction (4.1 $MPam^{0.5}$) than those measured for the basal (3.1 $MPam^{0.5}$) and prismatic directions (3.3 $MPam^{0.5}$). These findings were explained by the orientation dependence of slip system activations which provide a new way in the design of textured polycrystalline ZrB_2 ceramics towards enhanced damage tolerance.

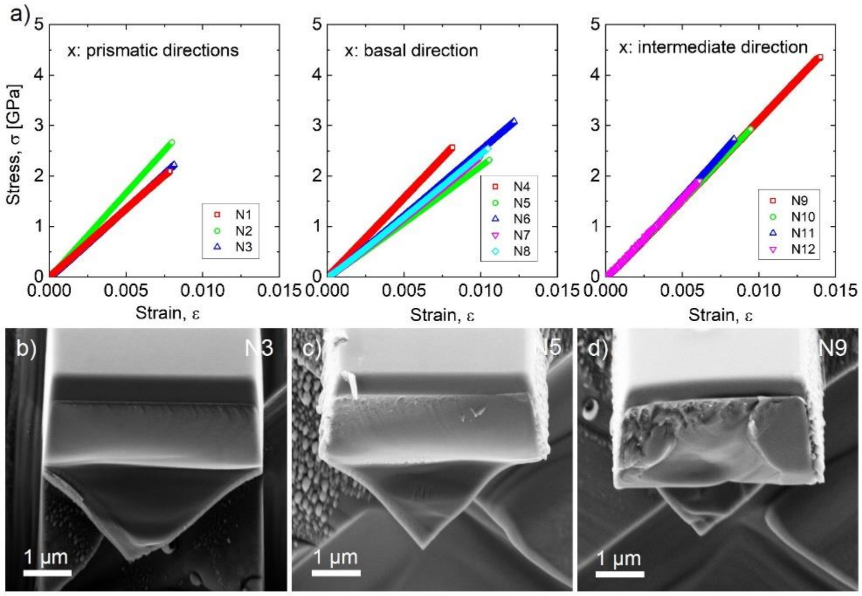

Fig. 1: a) The effective stress-strain curves for different orientations together with typical SEM images of the fracture.

Acknowledgements

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Scratch behavior of chemically tempered alkali borosilicate glass: measurements using Berkovich and conical indenter

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Keywords: Borosilicate glass, nanoindentation, scratch

Chemical tempering is a well-established method to enhance the mechanical performance of oxide glasses, such as bending strength; however, little is known about the scratch behavior of the surface of chemically tempered glasses. In the present work, a commercial alkali borosilicate glass was subjected to chemical tempering. The mechanical properties of the glass surface were determined by road-ramp scratch tests utilizing a Berkovich indenter in edge-forward orientation, producing sharp scratches, and a conical indenter for blunt scratching. While the grooves produced by the sharp indenter on the chemically tempered glasses exhibited a significantly larger recovery than the reference sample, the cracking and micro-abrasion behavior of the samples were similar. Surprisingly, the blunt indenter experiments revealed that chemical tempering decreases the onset of micro-cracking and wear abrasion of the borosilicate glass. Using a blunt indenter enabled the determination of the changes in the mechanical performance of chemically tempered glasses. The structural changes of glass structure due to chemical tempering, such as a decrease in free volume and the indenter-glass interaction, are responsible for the observed decrease in the onset of micro-cracking and wear abrasion.

Acknowledgement

This work was realized in FunGlass Centre founded from the European Union's Horizon 2020 research and innovation programme under grant agreement No 739566. Financial support of this work by VEGA 2/0028/21 is gratefully acknowledged.

Nanohardness, fracture resistance, and enhanced wear resistance of dual-phase high-entropy carbide/boride ceramics

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Keywords: high entropy carbide, high entropy boride, dual phase, ultra-high temperature ceramics, nanoindentation, wear

Dual-phase ultra-high temperature ceramics based on high entropy borides and carbides were prepared was prepared from powders synthesized via a boro-carbothermal reduction and sintered by spark plasma sintering. The resulting material was characterized with respect to its mechanical properties and wear behavior.

The material was a fine-grained dual-phase high-entropy (TiZrNbHfTa)C / $(TiZrNbHfTa)B_2$ - (HEC/HEB) composite. It showed very high density with a value of 8.72 g/cm^3 and consisted of small grain size of HEC and HEB grains with values of 0.95 ± 0.30 µm and 0.99 ± 0.27 µm, respectively.

The nano-hardness of the individual HEC and HEB grains was very high with mean values of 37.4 ± 2.3 GPa and 43.0 ± 2.9 GPa, respectively with the micro-hardness HV1 of the dual system as a whole being 29.4 ± 2.0 GPa. The material also possess improved crack propagation resistance thanks to promotion of intergranular crack propagation along the boride/carbide boundaries, and also due to crack branching inside the larger carbide grains.

Wear characteristics were investigated using the ball-on-flat technique/dry sliding in air. The friction coefficient values during the test with 5 N and 10 N increased from a value of 0.4 and reached up the values 0.65 and 0.77 at the sliding distances of approximately 1500 m and 1000 m, respectively. The specific wear rate decreased with increasing sliding distance at 5 N load, from 4.75×10^{-7} mm^3/Nm to 4.2×10^{-7} mm^3/Nm and at 10 N from 2.1×10^{-7} to 1.7×10^{-7} mm^3/Nm. The dominant wear mechanisms in both cases were an oxidation-driven tribo-chemical reaction and tribo-layer formation in boride grains and mechanical wear in carbide grains.

Acknowledgement

This work was done mainly in the frame of the project SASPRO2 No 1152/01/01 Dual phase high entropy ultra high temperature ceramics. The support of the Slovak Research and Development Agency under the contract no. APVV-22-0493 and of M-ERA.NET3/2021/82/DuplexCER/2022 is also greatly appreciated.

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Understanding Intrinsic Stress Effects on Vibrational Response of Silicon Nanowires

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Keywords: NEMS, Nanomechanical Modeling, Vibration, Silicon, Nanowire, Intrinsic Stress.

Silicon Nanowires (Si NWs) show significant promise as essential elements for advancing nanoelectromechanical systems (NEMS) and nanoelectronics, particularly in nano-resonators. The size-dependent mechanical properties of Si NWs pose a challenge for conventional continuum theories, requiring modifications for accurate predictions of their mechanical behaviors. The recently introduced ExtZP nanomechanical model [1] revealed up to 85 GPa variations in elastic properties of Si NWs, attributed to the model selection. This current study aims to propose a method for integrating intrinsic stresses (fabrication-induced and surface-inducted residual stresses) into continuum models to explore the flexural vibrational behavior of Si NWs. Fig. 1a illustrates a Si NW co-fabricated using a newly developed monolithic fabrication method [2]. A Laser Doppler Vibrometer (LDV) is employed for the high-precision characterization of vibrational motion of Si NWs. The analysis of Si NWs will explore frequency changes due to intrinsic stresses introduced via thermal processes, contributing to an improved understanding of intrinsic effects on their vibrational response.

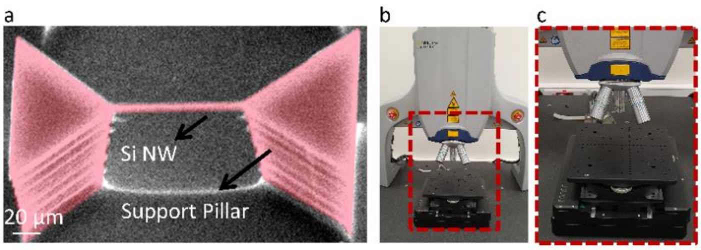

Fig. 1: (a) SEM image of a silicon nanowire (Si NW) and support pillars, (b) LDV Setup, (c) close-up.

Acknowledgement

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Understanding and validating the fracture behavior of damage tolerant diamond-metal laminates

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Keywords: Laminates, crack deflection, nanoindentation, toughness, flexural, fracture, bending, cohesive elements, composite, FEM.

Diamond metallic laminates (DML) have been demonstrated to exhibit an improved toughening mechanism by creating multiple opportunities for crack deflection through ductile interfaces. Laminates with two, five, and ten alternate layers of diamond and a metallic triple layer of sequence titanium, silver, and titanium were successfully produced by HFCVD and PVD. It was observed that the fracture propagates in a sequential step-like failure pattern under flexural loading. The current study aims to determine the contributing aspects and potential areas for improvement through analytical and experimental approaches. An array of micro cantilevers was realized with precision laser milling and tested their flexural properties with nanoindentation. The difference with the unnotched to the notched laminates made us understand the fracture sensitivity on overall stiffness of the composite. The results are promising in a way and comparable to the bulk 3-point bending approach. A 2D model of FEM with cohesive elements was designed in same experimental scenarios (3-point bending, micro cantilever bending) to understand the diamond-metal interfacial properties, crack propagation etc. The outcome from the model and experiment are in good agreement and paved a way to optimizing the composite's overall design in order to increase the toughness.

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Posters

Multi-parameter optimization of layered WS_2-polymer nanocomposite under mechanical loading

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Keywords: tungsten disulfide, analytical modeling, multi-parameter optimization, nanocomposite geometry, safety load

In the present study, the influence of the geometry at nano and micro level (layer thicknesses and length) and the magnitude of axially applied mechanical load on the delamination in three-layer WS_2-polymer nanocomposite, is theoretically investigated. First, the analytical solutions for the interface shear stress (ISS) in the middle layer of the structure are obtained, based on the application of 2D stress-function method and minimization of the strain energy [1]. Second, the theoretical criterion for delamination in the interface layer (non-linear equation in terms of debond length), based on the ISS model is formulated, including the structure geometry and loading as parameters. Third, a multi-parameter optimization problem including this criterion is defined and solved. By simultaneously varying these parameters, the safety intervals of the parameters (without delamination) in the considered nanocomposite structure are obtained. It was found, that the magnitude of the applied load mainly affects the magnitude of the ISS. Layers thicknesses mostly affect the type of ISS solution, especially the substrate thickness. The effect of layer length on ISS is weaker than that of layer thickness at a fixed load. The obtained results can be used for fast prediction of delamination and appropriate design in similar nanostructured devices to assure their safety work.

Acknowledgement

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Parametric analysis for interface shear stress in MoS_2/PET nanocomposite under thermo-mechanical loading

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Keywords: molybdenum disulfide, parametric analysis, interface shear stress, nanocomposite, thermo-mechanical loading

In this study, a parametric analysis on the factors, that influence the value of theoretical interface shear stress (ISS) in nanocomposite MoS_2/PET [1], subjected to thermo-mechanical loading has been performed. The theoretical value of ISS is calculated from the already obtained analytical solutions for the interfacial shear stress in our previous works [2]. The sensitivity of following parameters on the ISS in the considered nanostructure is investigated: the thicknesses of the nanocomposite layers, the length of the nanocomposite, the magnitude of the applied mechanical load, the applied temperature difference. It is found that the interface thickness does not affect the ISS value. The magnitude of the applied mechanical load has a strong influence on the magnitude of the ISS. The temperature difference (pure thermal loading) also affects the ISS value, but to a lesser extent. It should be noted, that in the case of combined loading (thermo-mechanical) the overall effect is additive. The thicknesses of MoS_2 and PET mostly affect the type of ISS solution, especially the substrate thickness. The obtained results are graphically illustrated and can be used for fast prediction of ISS in micro scale in similar nanocomposite devices or parts thereof such as sensors, nano- and optical electronic devices, energy devices, etc.

Acknowledgement

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Addition of dental fillings with nanoparticles to improve their mechanical properties

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Keywords: glass ionomer cement, nanoparticles, mechanical and physical properties, tribological properties

Glass ionomer cement (GICs) have gained much attention in the second half of the last century, especially in the field of dental medicine. They have attracted attention mainly due to their antibacterial properties, ability to release fluoride, and partial remineralization of dental tissue. Unfortunately, despite its considerable advantages, GIC is mainly used for the treatment of deciduous teeth due to insufficient mechanical and physical properties (deficiencies in mechanical properties and wear resistance limit their applicability).

This research focuses on modifying dental restorations, specifically glass ionomer cement, to improve its mechanical performance properties. This work aimed to use nanoparticles (zinc oxide or nanodiamonds) to modify a commercially used glass ionomer cement to improve the material's mechanical-physical properties (adhesion, hardness, strength, abrasion resistance).

Glass ionomer cement is formed by mixing two main components, powder and liquid. The powder contains finely ground silica glass, calcium, aluminum, and fluorine compounds. The liquid is polyacrylic acid. By mixing these two components in precise proportions, an acid-base reaction occurs, resulting in the formation of a solid material. For the purpose of our research, zinc oxide nanoparticles, diamond nanoparticles, and hydroxylated modified nanodiamonds were selected at concentrations of 0.25 wt% and 0.50 wt%. The nanoparticles were added to the powdered portion of glass ionomer cement in a pre-calculated weight to match the given concentration. The size and shape of the samples were determined according to the specifications for each measurement.

This study reviews two types of nanoparticles used to modify dental restorations and their effect on the material's adhesion, hardness, strength, and wear resistance. Emphasis is placed on the significant potential of nanoparticles for improving the mechanical properties of dental restorations, reducing sensitivity, and preventing caries recurrence. The results of these innovations suggest that modifying dental fillings with nanoparticles can bring about changes in the field of dentistry and provide patients with more durable and effective dental fillings.

Acknowledgement

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Effect of selected HVOF-sprayed coatings parameters on dynamic impact wear

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Keywords: HVOF, impact test, impact wear, microstructure, roughness

High-velocity oxygen fuel (HVOF) sprayed coatings are widely used in industry due to their corrosion resistance, chemical stability or high-temperature resistance. In addition, some of them have to resist particle impact or dynamic interactions with components. However, these dynamic interactions reduce the coating lifetime [1]. In order to optimize the coating lifetime, it is necessary to know the response of HOVF sprayed coatings to dynamic impact loads.

Dynamic impact load was investigated using a dynamic impact tester. The tester repeatedly impacted the same place on the coatings surface with constant frequency and constant impact load. The impact lifetime was estimated from the residual impact crater parameters using a modified Engel model [2]. The effect of repeated impact on the microstructure of the coating was studied using the cross section under the impact craters.

Impact analysis of the HVOF-sprayed Cr_3C_2-25%NiCr coatings revealed strong dependency of the impact lifetime on the coating thickness (examples of cross sections are shown in figure 1). The effect of sample surface roughness and substrate material on impact wear was also discussed.

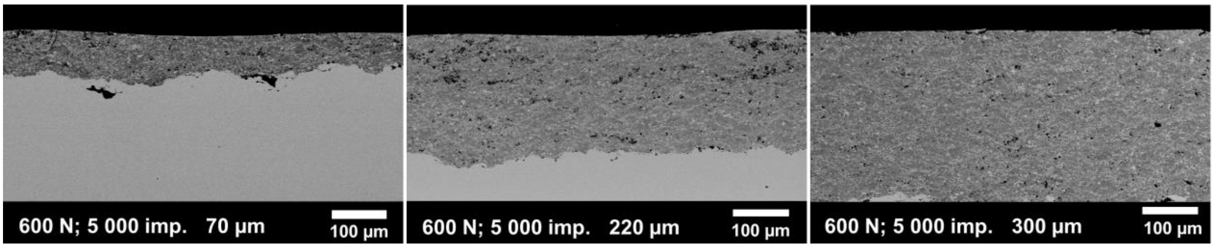

Fig. 1: Comparison of impact craters cross sections.

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Utilization of the Shear Test to measure the Local Mechanical Properties of Duplex Steels Welds

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Keywords: welding, shear test, duplex steel

The quality criterion of a weld joint is its integrity, geometry and mechanical properties. Hardness, strength and toughness are the most frequently tested properties of weld joints determined by testing. The mechanical characteristics of joints are a quantitative expression of mechanical properties. During the microhardness test, it is possible to measure the hardness in all parts of the weld joint, which consists of the base material, the heat-affected zone and the weld metal. During the toughness test, the toughness can be measured in all parts of the weld joint by selecting a suitable sample and positioning the notch. In the tensile strength test it is more complicated. The sample breaks at the point of least strength or at the point of the defect. If there is a fracture of the basic material, the joint is evaluated as satisfactory, but we do not get an overview of the strength of the individual parts of the weld joint. It is not possible to measure local mechanical properties - the strength in a predetermined part of the weld joint. Therefore, it is necessary to choose another method for measuring this property in any predetermined part of the weld joint, to be able to determine the strength of butt weld joints in the base material, in different areas of the heat affected zone, in the weld metal. Some of such methods are based on tensile testing of miniature test specimens prepared from the desired weld joint area with a gauge length of several millimeters [1] or even fractions of a millimeter in the case of small welds [2]. These experimental methods are experimentally and apparatus very demanding and more difficult to apply. Therefore, a method of measuring local mechanical properties of welds by shear test was developed. It would be possible to measure shear strength in butt welds of base materials, in different places of the heat affected zone, in weld metal. The method was applied to the measurement of the weld strength duplex steels.

Acknowledgement

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Influence of Laser Beam Welding Parameters on Local Mechanical Properties of Duplex Stainless Steel Joints

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Keywords: welding, welding parameters, weld joint, laser, duplex stainless steel, shear test

Duplex stainless steels (DSS) are dual-phase alloys consisting of austenitic and ferritic phases in the microstructure. DSS provide an ideal compromise between mechanical properties and corrosion resistance. Welding of DSS leads to a change in the ferrite/austenite ratio in the microstructure and to degradation of the properties of the weld joints. Therefore, it is very important to control the microstructure of the weld joint directly during the welding process. An innovative method of laser welding applying a dual laser beam was used. By spreading the energy of the laser beam over two spots, additional heat is introduced into the weld, which ensures a slowdown in the cooling rate of the DSS and ensures the desired ratio of ferrite and austenite in the microstructure. The influence of laser welding parameters and energy distribution of the dual laser beam on the resulting microstructure and local mechanical properties of the weld joints was monitored. During the tensile strength test, the sample is broken at the area of least strength or defect. This does not give an overview of the strength of individual parts of the weld joint, such as the base material, heat-affected zone and weld metal. The strength properties were therefore assessed by applying a shear strength test in a predetermined area of the weld joint. Samples of weld joints were subjected to visual inspection, metallographic analysis, microhardness measurement and local shear strength test. The goal was the design and optimization of welding parameters in order to achieve satisfactory properties of weld joints.

Acknowledgement

This work was supported by the Slovak Research and Development Agency under the contract No. APVV-21-0232 and under the contract No. APVV-23-0134.

About the choice of the indenter to determine mechanical properties of superalloys by using high temperature microhardness tester

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Keywords: high temperature, microhardness, superalloy, mechanical resistance

In the aeronautical field, materials are used in severe environmental conditions (temperature, atmosphere), particularly for engine applications. In order to qualify new alloys compositions, ONERA performs micro-indentation tests from room temperature up to 750°C close to operating conditions. This method presents the interest of performing tests faster than classical mechanical tests like tensile or bending tests under severe conditions.

Nevertheless, the choice of the indenter for temperature applications is the most important choice [1]. The use of diamond, sapphire, tungsten carbide, silicon carbide, boron carbide, boron nitride to make indenter are discussed and compared. Characterizations by using electronic microscopy, X analysis, and ATG of most interesting indenters for our application are presented.

Finally, tungsten carbide indenter seems to be the best indenter for using it to characterize Nickel base superalloys. An experimental method is proposed to control the evolution of the indenter during its use in the microhardness scale.

Determination and evolution of mechanical resistance versus temperature by indentation tests by using Tabor relationship by using tungsten carbide indenter are discussed by performing tests on a Nickel based superalloy and are compared to mechanical resistance values determined by tensile tests.

In perspective, these hardness measurements could be carried out up to 1000°C if indenter is still available. Many other applications on different materials such as layers, coatings, composite materials, brazing cords or additive manufacturing materials seems to be atteignable.

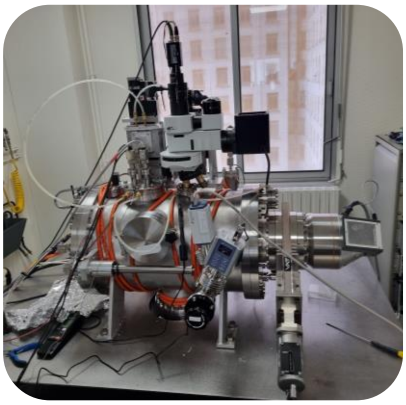

Fig.1: High temperature micro hardness prototype developed by ONERA

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Efficient mapping of mechanical properties using Gaussian processes

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Keywords: mechanical properties mapping, instrumented indentation, Gaussian processes

Detailed mapping of (not only) mechanical properties using techniques such as instrumented indentation or scanning probe microscopy techniques, can be a very lengthy process, however, acquiring the complete image might not be needed e.g. when looking for a specific feature. We present a custom software tool, which, when combined with a suitable measuring instrument, enables to quickly identify regions containing possible anomalies, and to gather information from suspect regions in the first place.

The foundation for the tool is provided by iteratively employing Gaussian processes (GP), which can be considered a form of machine learning, and can be applied as an interpolation tool, able to predict the function value as well as the variance (uncertainty) of the prediction at a given point. GP can be implemented in computer using standard tools for linear algebra and optimization without need for large computation toolboxes.

With our tool in each iteration, a GP is constructed and trained using data already acquired, and then used to suggest unmeasured points where uncertainty of the prediction is greatest – this typically corresponds to areas in which the measured quantity changes noticeably. Performing the measurement at the suggested points provides quick reduction of the overall uncertainty on the whole unmapped area. The procedure is then repeated until sufficient amount of information is gathered.

Acknowledgement

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Modulus estimation of polymers via nanoindentation – impact of surface roughness and peak force

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Keywords: polymer, nanoindentation, roughness

Nanoindentation is a powerful tool, to measure mechanical properties of even the thinnest foils and layers. However, a profound understanding of the mechanical behavior of the sample is necessary to evaluate the results. The influences which have to be considered are manifold. One of the main influences of indentation tests, the surface roughness, is the focus of this study. The aim is to show the decisive influence of the roughness on the necessary number of indentations to get trustworthy results, as well as the possibility to overcome roughness induced measurement errors with higher applied forces. As test material, commercially available polyethylene (PE) was chosen, since the mechanical properties are well known. The sample roughness was obtained through grinding with sandpaper of varying grain sizes and polishing. Due to the softness of PE, different methods of roughness measurements were compared. Confocal laser microscopy proofed itself as reproducible and reliable. For the indentation tests a Berkovich Indenter was chosen, due to the sensibility to roughness. Figure 1 shows that the lower roughness results in measurements near the expected 1.4 GPa, while the rougher surface needs much higher peak forces to deliver the same result.

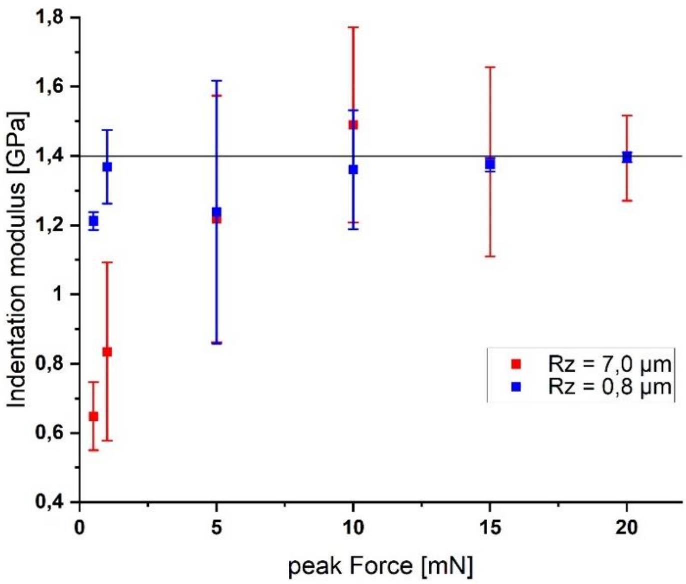

Fig. 1: Influence of surface roughness on the necessary peak force for correct stiffness evaluation.

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The research work was performed at the Montanuniversitaet Leoben within the COMET-project 3.S4 of the Polymer Competence Center Leoben GmbH (PCCL, Austria) within the framework of the COMET-program of the Federal Ministry for Transport, Innovation and Technology and the Federal Ministry for Digital and Economic Affairs. The PCCL is funded by the Austrian Government and the State Governments of Styria, Lower Austria and Upper Austria.

Nanoindentation analysis of high fluence helium ion irradiated Eurofer 97 and ODS Eurofer steels

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Keywords: nanoindentation, ion implantation, radiation resistance, helium transmutation, simulation

Addressing the challenge posed by elevated levels of transmutation helium in materials for use in advanced nuclear reactors both fusion and fission is crucial, as it leads to the formation of helium bubbles, causing embrittlement and swelling [1]. While current research primarily examines fundamental aspects such as defect evolution and void swelling at the nanoscale, our study particularly investigates the bulk properties of these materials and their practical engineering applications.

To study the effects of transmutation helium, we subjected Eurofer97 and its ODS variant, as well as five other structural steels—SIMP, PM2000, OSD Eurofer, SS 310S, and 800H—designed for use in demanding radiation environments, to irradiation. This study homes in on the mechanical characteristics of Eurofer97 and its ODS counterpart. Using a helium ion beam with energies ranging from 17 MeV to 1 MeV, we systematically decreased the energy levels to create a uniform "box-profile" with a helium concentration of 1000 appm [2]. This method resulted in a consistently irradiated layer approximately 65 µm thick, which enabled subsequent micromechanical testing and the assessment of engineering-relevant (bulk) material properties.

In this contribution, we present the initial findings of our work, particularly nanoindentation analysis of mechanical properties of the irradiated steel properties.

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Mechanical Properties of Cold-Sprayed Ti-6Al-4V Coatings on Al 7075 Alloy

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Keywords: Ti-6Al-4V, cold spraying, microstructure, nanoindentation

POSTER PRESENTATION

The objective of the presented studies was to analyze the microstructure and mechanical properties of a cold sprayed Ti-6Al-4V structure for application in additive manufacturing. The coatings were sprayed using the Impact Innovations 5/8 system with the robot Fanuc M-20iA at Kielce University of Technology. The feedstock used for this study was commercial Ti-6Al-4V powder with a "coral-like" morphology. The working gases used in this process were nitrogen and helium in equal proportions. The coatings sprayed onto the titanium mandrel had a thickness of 15 mm. The microstructure and chemical composition of the powder and the coating were analyzed by means of SEM Jeol JSM-7100, and their phase composition was studied using a Bruker D8 Discover diffractometer. The micromechanical testing of coatings was carried out with the use of the nanoindentation technique (Nanovea) with a Berkovitz indenter (the Olivier and Pharr methodology). The high kinetic energy of feedstock particles and their morphology caused significant deformation, and particular splats strongly adhered to the substrate and to each other. Throughout the cross-section, the coating was homogenous and exhibited negligible porosity. On the other hand, histograms and probability distributions of the hardness and elastic modulus of cold sprayed coating showed significant differences (Fig.1).

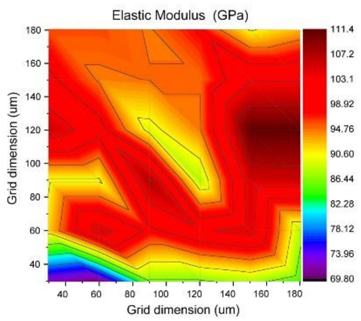

Fig. 1: Distribution of elastic modulus for cold-sprayed Ti-6Al-4V coating.

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A nanoindentation study of TiN films deposited using magnetron sputtering under various condition

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Keywords: titanium nitride, ceramics, thin films, nanoindentation, scratch test

Titanium nitride (TiN) films are widely used for various purposes, notably as a protective layers for highly stressed drills or mills due to their mechanical properties, corrosion resistance and even for their aesthetical properties . As member of conductive nitride family it is thoroughly studied since the 1930s in bulk form and from 1970s in thin film form thus being nowadays regarded as one of the most important technological materials [1]. Its prevalence has also been supported by the suitability of production using a range of chemical vapor deposition (CVD) and physical vapor deposition techniques (PVD) [2, 3]. The latter includes magnetron sputtering and its variants, which allows precise control of the production, i.e. deposition process and achieving the required stoichiometry, thickness and structure. Several studies focused on investigating the effect of deposition conditions on the mechanical properties were already published [4-6], however with the expansion of contemporary processes such as HiPIMS [6], comparative studies with fine-step parameter changes within a well-reproducible are highly desirable.

In this research two main groups of TiN thin fims were deposited on silicon substrates using reactive magnetron sputtering operating in pulsed DC mode and in HiPIMS mode. Nitrogen content (flow rate) varied while argon was kept constant, similarly as other deposition parameters like pressure, power and deposition time. Resulting films were studied using nanoindentation and scratch test to obtain mechanical properties (hardness, reduced modulus) and adhesive-cohesive properties. Scratch test was evaluated using standard techniques (microscopical imaging and depth change record) upgraded by simultaneous detection of acoustic emissions. Nanoindentation results show an increase in mechanical properties with nitrogen concentration in both groups, however after reaching the highest values, the hardness and modulus of elasticity started to stagnate or slightly decline. Highest hardness was achieved for N_2/Ar flow rate ratio 0.133 for pulsed DC and 0.033 for HiPIMS. In terms of attrition tests, the highest differences can be observed for low nitrogen concentrations, when ductile layer removal occurs. At higher concentrations, more brittle damage modes appear which are delayed in some samples. Thus, in terms of scratch resistance, samples with N_2/Ar flowrates of 0.267 - 0.400 for Pulsed DC and 0.067 - 0.133 for HiPIMS appear to be slightly more resistant.

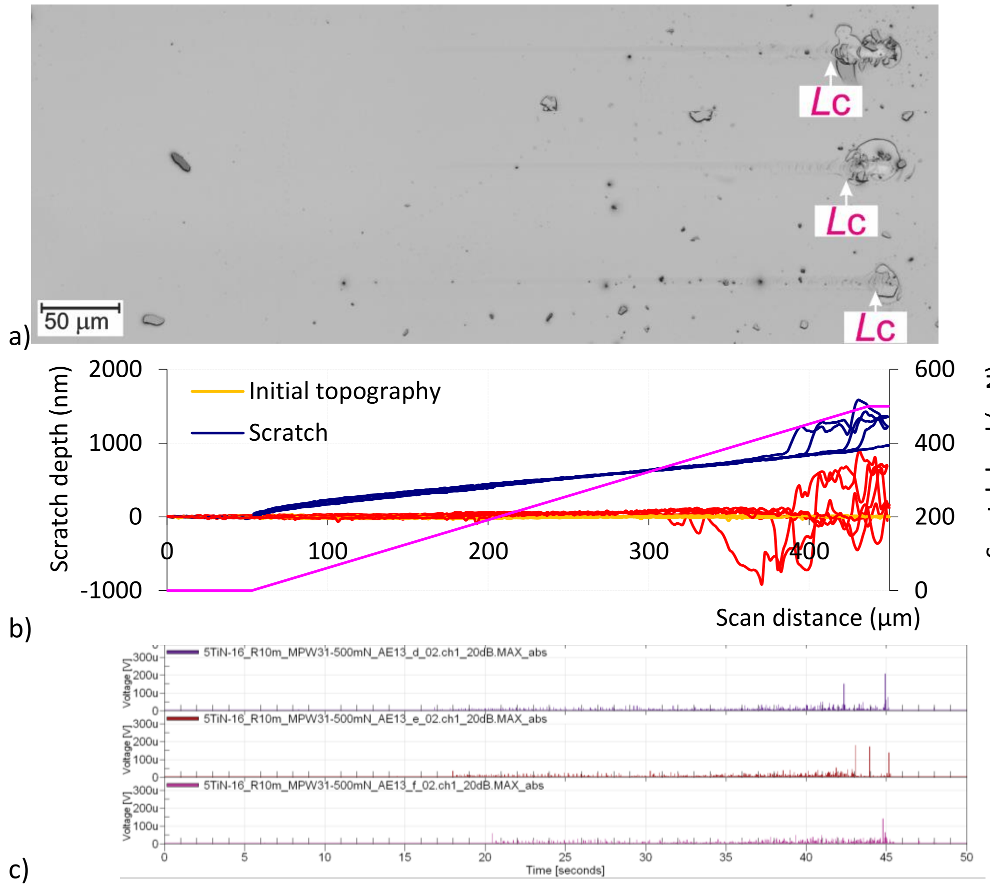

Fig. 1: Example of scratch test evaluation of TiN-16 sample using three independent methods a) microscopic evaluation, b) depth change and c) simultaneous detection of acoustic emissions.

Acknowledgement

The authors gratefully acknowledge the support by the project IGA_PrF_2023_005 of Palacky University. This work was co-financed with M-ERA.NET Technology Agency of the Czech Republic (No. TA ČR TH80020005) and partner projects grant of the Romanian National Authority for Scientific Research and Innovation, CCCDI – UEFISCDI, project number COFUND-M-ERANET-3-HardCoat-1, no. 311/2022 (INOE), within PNCDI III and tax revenues based on the budget approved by the members of the Saxon State Parliament.

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A new approach to the properties of matter in terms of nanoindentation research on the example of selected materials

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Keywords: nanoindentation, cold spraying, metals

Nanoindentation research and modern software allow for expanding the possibilities of examining the properties of matter. The paper presents measurements of hardness, elasticity modulus and adhesion of coatings on a titin substrate. Coatings applied using the cold spray technique. The following materials were used as coating materials: copper, titanium, brass and magnesium.

The hardness and elastic modulus were calculated from the shape of the load and unload curve. A difference in the shape of the unload curve was noticed. A coefficient was introduced reflecting the symmetries of the curves $W = (W_I-W_M)/W_I$ and called the workload of the sample, where W_I, W_M are the area under the load and unload curve, respectively.

The correlation between the modulus of elasticity and the W coefficient was examined. Figure 1 shows the surfaces used for W calculations

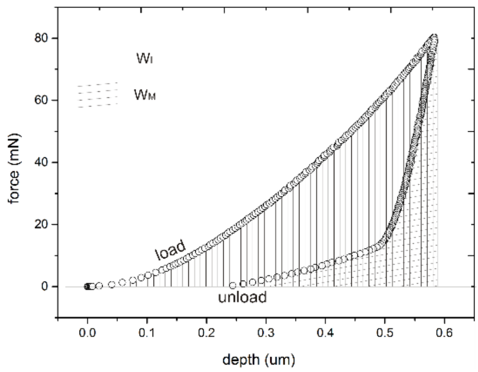

Fig. 1: Example of load and nuload curves with indication of the area.

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Mechanical and thermal reinforcement of the polymer based graphene nanoribbon composites

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Keywords: nanocomposites, graphene nanoribbons, mechanical and thermal reinforcement

The mechanical and thermal reinforcement of the nanocomposites from graphene nanoribbons (GNRs) and poly(methyl methacrylate co- butyl acrylate-2-hydroxyethyl methacrylate) was analyzed with TA.HD plus texture analyzer and dynamical mechanical thermal analyzer.

The morphology of the nanocomposite films was determined using scanning electron microscopy, where the samples were scanned without sputtering with metal. Two morphologies of the nanocomposites were developed, one where the GNRs are mostly randomly oriented, characteristic for the lower GNRs loadings, and, the other for the nanocomposite with 3.0 wt %, where the GNR encapsulated the polymer particles.

The effect of adding GNRs on the mechanical properties of the nanocomposites measured by tensile test is in the direction of the important reinforcement achieved compared to the neat polymer film in terms of increased Young's modules, offset yield stress and hardness, with a drop in the elongation at brake, typical for reinforced composites. The reinforcement is proportional to the GNR loading. The Young's modulus of elasticity was increased up to 66 fold with respect to that of the neat polymer film (for 3% GNR), whereas the 9 fold augmentation is observed for the offset yield stress.

The elongation on the other hand was almost 2 fold lower. This means that the nanocomposites are stiffer than the neat polymer due to the presence of GNRs. Namely, GNRs act as load transfer centers, assisting in the applied stress transfer from the polymer to the nanoribbons, providing more strength to the nanocomposites to endure the applied stress. Much lower effect to the elongation at brake proves that simultaneously the elasticity was preserved, which effect is not usual for graphene based fillers. This effect may be explained by the elasto-plastic behavior of GNRs and their high flexibility. Actually, GNRs likely act as a bridge across the cracks absorbing the fracture energy, which ensures high flexibility along with improved stiffness.

The good interfacial strength between the polymer and GNRs providing good stress transfer through the nanocomposites indicates that the interface polymer – GNR is characterized by establishing strong interactions between both components.

The obtained nanocomposites are distinguished with improved mechanical and thermal properties; owing to the good stress transfer through the nanocomposites.

Changing the integrity of the material surface by combining Laser Surface Texturing and PVD Magnetron Sputtering technologies

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Keywords: PVD magnetron sputtering; laser micromachining; mechanical and tribological properties

The presented research combines laser micromachining technology (LST, Laser Surface Texturing) with an ultra-short femtosecond laser beam and PVD deposition of thin films using magnetron sputtering.

This study aims to investigate the advantages of combining laser technology with magnetron sputtering and evaluate the synergistic effects with a focus on improving the tribological properties of surfaces.

Modern femtosecond lasers, used in LST technology, have become a key tool in precisely affecting the surface of materials with minimal thermal damage. The technology is chosen to create various LST surface textures, including forming a sub-microrelief called LIPSS (Laser-Induced Periodic Surface Structure). Laser texturing (Fig. 1), combined with magnetron sputtering, significantly improves the useful properties and preserves the integrity of the material.

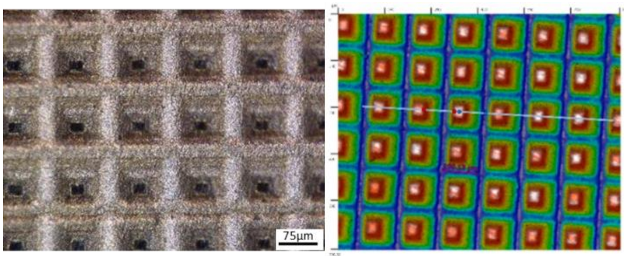

Fig. 1: Visualization of the texture on the sample surface

Magnetron sputtering adds another dimension to improving the surface properties of the material. This coating technology enables the formation of homogeneous thin layers with high adhesion to the base material. Sputtering, in combination with LIPSS textures, produces uniform coverage of the thin film and ensures good integration of the new coatings with the micro-reliefs created by the LST technique.

This synergy between technologies (femtosecond laser and magnetron sputtering) provides advantages in the form of homogeneous and high adhesive coatings with better tribological properties. The results of this study will contribute to a broader understanding of the possibilities of combining these technologies and their importance in the field of surface engineering and materials research.

Acknowledgement

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Finite element analysis of the pile-up and correction of projected contact area

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Keywords: finite element method, nanoindentation, pile-up, parabolical correction, steel

This paper deals with pile-up, which can occur at nanoindentation. FE simulation of X5CrNiCuNb16-4 steel nanoindentation was done and stress and strain causing pile-up behavior were analyzed in more detail. Pile-up also influences projected contact area, which should be corrected to include the pile-up into Oliver-Pharr analysis [1]. To precisely calculate the projected contact area, its border has to be known. Few methods of border approximation were compared, specifically approximation with the triangle [2] and the semi-ellipse [3]. For more precise approximation, the expression for parabolical approximation was derived. All methods were compared with the projected contact area calculated by FEM (Fig. 1). The most precise results were obtained with the semi-elliptical and parabolical correction and can be used for determination of the projected contact area and its borders.

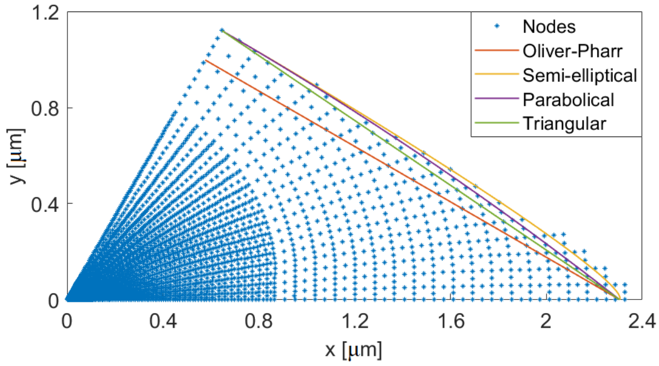

Fig. 1: Comparison of the projected contact areas of the 1/6 of Berkovich indenter imprint

Acknowledgement

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Solid solution strengthening of Zn-based alloys measured by micro-pillar compression

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Keywords: zinc alloys, micro-pillar compression, critical resolved shear stresses

Due to their biocompatibility and degradation properties, Zn alloys are emerging as promising candidates for temporary biodegradable metallic implants, such as bone-fixing plates or cardiovascular stents. However, the relatively low mechanical strength of conventional Zn alloys has historically led to limited research interest in the deformation mechanisms of fine-grained Zn alloys.

This presentation will explore these mechanisms, particularly highlighting the impact of solid solution strengthening and the size effect observed through micro-pillar compression tests and post-mortem EBSD analysis of deformed pillars. We will also delve into the role of grain boundary sliding and slip system activity in the superplasticity of bulk Zn alloys, with these phenomena being studied across a broad spectrum of strain rates using a combination of EBSD-SEM-AFM techniques. The investigation provides comprehensive insights into the effectiveness of solid solution strengthening and the primary deformation mechanisms in fine-grained, single-phase Zn alloys at room temperature.

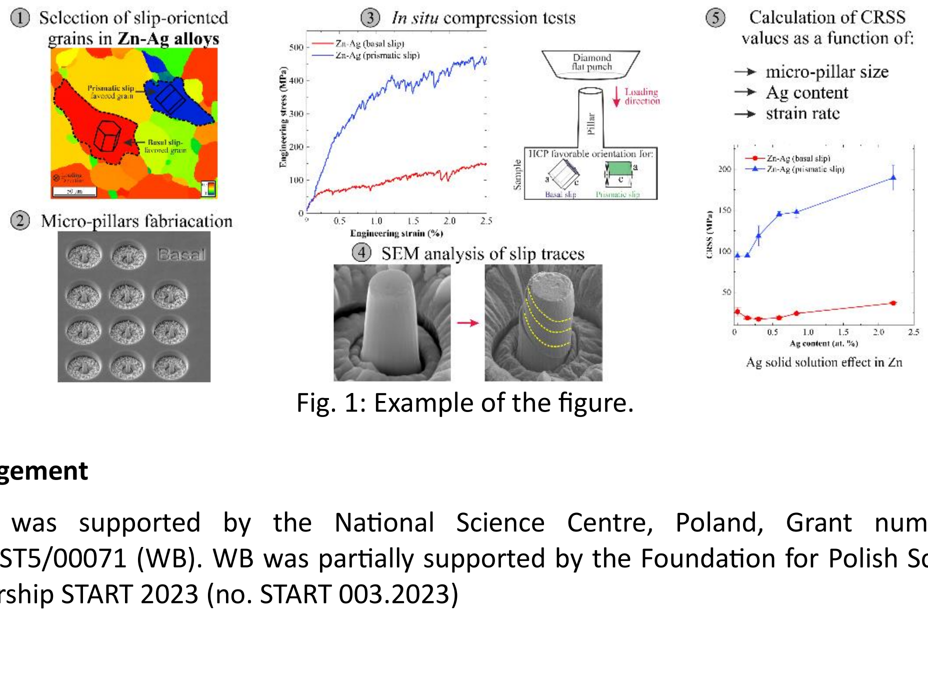

Fig. 1: Example of the figure.

Acknowledgement

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Nanoindentation creep behavior of UHMWPE and PEEK polymers used in total joint replacements

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Keywords: nanoindentation creep, viscoelastic, polymers, total joint replacements

The investigation of polymers with nanoindentation (NI) is challenging since the deformation during loading is time-dependent, which can be described by viscoelasticity. Creep experiments can be combined with viscoelastic material modelling to better understand the time-dependent material behavior of polymers. In this study, spring-dashpot models will be used to describe the viscoelastic behaviour of three synthetic polymers: virgin ultrahigh molecular weight polyethylene (UHMWPE), highly-crosslinked UHMWPE (UHMWPE-XL) and polyether-ether ketone (PEEK). These polymers are employed as liners in total joint replacements (TJR) in orthopedic surgery. Their mechanical performance is one of the factors influencing the TJR lifespan [1].

In former works the influence of semi-crystallinity on NI creep results obtained for polyoxymethylene (POM) [2] in comparison to amorphous poly(methyl methacrylate) (PMMA) [3] was investigated by modelling with the general Maxwell model, as this is a common approach for viscoelastic creep modelling. Here it turned out that the general maxwell model may be too simple to describe viscoelastic behaviour of a semi-crystalline polymer. Therefore this study extends the work on semi-crystalline polymers with UHMWPE, UHMWPE-XL and PEEK as role-models, by the use of freeware Python package MCREEP [4, 5] for viscoelastic modelling. The package can fit more complex models to indentation creep data in a well-defined, consistent, and user-friendly way. This enables us not only to describe the creep behavior, but also to compare the performance of models at all length scales, and to predict the long-term creep properties.

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Estimation of Mechanical Properties of Neutron-Irradiated 08Ch18N10T Steel from Hardness Testing

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Keywords: 08Ch18N10T steel, radiation embrittlement, nuclear reactor internals, Vickers hardness, yield strength, finite element simulation

Through-thickness Vickers hardness of a neutron-irradiated small segment of the core barrel of the WWER-440 type nuclear reactor made from 08Ch18N10T austenitic stainless steel was characterized experimentally. Results were compared to ones simulated numerically with assumption of Ludwik's form of hardening equation and neutron dose dependent material parameters according to work of Sorokin at al [1]. Simulated values of hardness trace the lower bound of scattered experimental data. Based on the same material input data and on the correlation with measured hardness profile, increase of the material yield strength of the core barrel due to reactor operation was estimated. Predicted yield strength in Fig. 1 decreases from 700 MPa close to inner surface of the barrel to 660 MPa 20–25 mm under the inner surface.

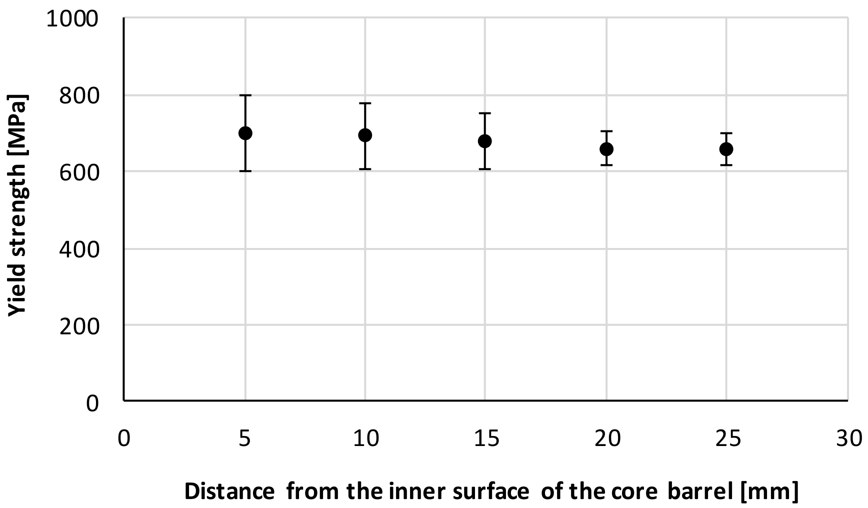

Fig. 1: Estimated yield strength through the wall of the core barrel.

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AFM-in-SEM: Understanding mechanical properties of low dimensional materials

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Keywords: AFM-in-SEM, nanostructures, Young modulus, mechanical properties, force-distance spectroscopy

Understanding the mechanical properties of materials often necessitates a comprehensive insight into their local properties at the nanoscale. This includes not only mechanical characteristics but also a complex characterization of the shape, size, surface topography, roughness, and material composition. These factors contribute to the diversity in mechanical properties, making it crucial to identify the origins of such behaviors.

In this study, we explored the mechanical properties of spider silk nanowires. These protein-based elongated micro- and nanostructures have potential for various biomedical applications, as surface functionalization and carriers for drug delivery. The new method of protein production is evolving, as it currently lacks precise control of either shape and dimensions or renders structures fixed to substrates [1]. The objective was to determine the elastic and plastic deformation of the nanowires suspended between the pillars. We utilized the spectroscopy mode of AFM LiteScope in SEM MIRA 3XMU, where we precisely located and navigated the nanowire, approached its surface, and measured force-distance characteristics using a three-point bending test, see Fig. 1. We were also able to determine the size and shape of the released protein using AFM topography, which further enabled us to calculate the Young's modulus of these unique nanostructures.

Thus, the AFM-in-SEM instrumentation approach plays a pivotal role in the characterization of mechanical properties of such nanostructures that are otherwise exceedingly difficult to observe.

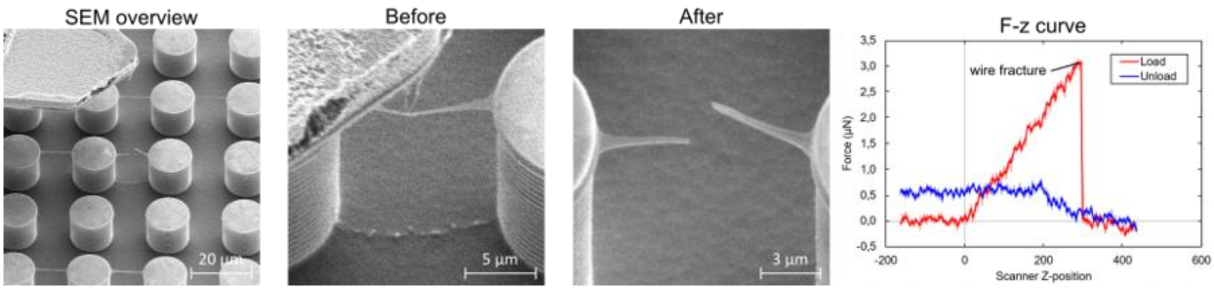

Fig. 1: Mechanical characterization of nanowires by AFM-in-SEM.

Acknowledgement

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Nano- and micromechanical testing of helium implanted reactor materials

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Keywords: ODS steel, nanoindentation, micropillar compression, ion-irradiation, helium embrittlement

Ion irradiation has proved to be a suitable surrogate for neutron irradiation enabling study fundamental radiation effects on materials [1-3]. It has been used during the last decades in fission and fusion reactors materials research to accelerate exposure. Ion-implantation enables much faster damage accumulation, achieving doses equivalent to several years of neutron irradiation in just a matter of hours or days. This approach presents several additional benefits: controlled experimental conditions, dose flexibility, reduced activation of samples and radioactive waste. However, one of the drawbacks, compared to neutrons, is the limited penetration of the accelerated ions into the material. Using low-energy protons (1-2 MeV) or heavy ions (at 2-5 MeV), commonly available in ion irradiation facilities for materials research, the depth of damage is typically from submicron to a few micrometers in common reactor materials. Furthermore, the dose rate varies with penetration depth.

The localization of the damage within a shallow near surface region poses challenges on the extraction of mechanical properties of ion-irradiated materials. Small-scale mechanical testing is often limited to nanoindentation. The interpretation of nanoindentation data is not straightforward, as the results are convoluted with the substrate effect and the size effect, and the damage level varies across the thickness of the irradiated layer.

In the present study nano- and micromechanical testing is performed on perspective reactor steels and ODS steels which undergone a dedicated irradiation process. Leveraging the ion energy range provided by the 6MV tandem accelerator at ATRI (ranging from 0.5 to 18 MeV for Helium), a multi-step ion-irradiation process was designed. This approach aims to create a thick, roughly 60 µm layer with uniform helium concentration and radiation damage (dpa) [4]. The substantial thickness of this layer offers the opportunity to employ micropillar compression techniques, alongside surface and cross-sectional nanoindentation. The aim of the work was to study the uniformity of the irradiated layers and the effect of transmutational helium on the mechanical properties of the selected reactor materials.

Acknowledgement

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