EXPERIMENTAL FRACTURE MECHANICS AT THE MICRON SCALE: FUNDAMENTALS, CHALLENGES AND PITFALLS

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NANOSCALE RESIDUAL STRESS AND ADHESION ASSESSMENT

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NEW ELECTRON MICROSCOPY TECHNIQUES FOR DETERMINATION OF LOCAL STRUCTURAL FEATURES DURING PLASTIC DEFORMATION

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Key Words: Electron Microscopy, Nanomechanics, In situ, High Entropy Alloys, Ti-Al.

This talk will highlight recent advances in Transmission Electron Microscopy (TEM) techniques that provide insight into small-scale plasticity and the evolution of defect structures in materials. Through the development of fast direct electron detectors, it is now possible to acquire large multidimensional data sets of nanodiffraction patterns (4DSTEM) that can map local structural order and strain with nanometer precision, even during in situ nanomechanical testing. The method is widely applicable and examples will be given from systems such as organic semiconductor molecular thin films, structural alloys with local order such as Ti-Al and CrCoNi, and even to amorphous samples such as bulk metallic glass. This talk will describe our recent results utilizing fast direct electron detectors, energy filtered imaging and in situ TEM nanomechanical testing that provide insight into multiscale materials phenomena using these techniques.

RECENT PROGRESSES IN IN-SITU AND 3D HR-EBSD TECHNIQUES TO ASSESS DEFORMATION MECHANISMS OF MATERIALS AT SMALL SCALE

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Key Words: HR-EBSD, tensile testing, twinning, magnesium, fracture mechanics, tungsten

Understanding deformation mechanisms of materials at the sub-micron scale requires advance characterization techniques capable of measuring microstructural changes, strains, stresses and lattice defects evolution during deformation. High angular resolution electron backscatter diffraction technique (HR-EBSD), coupled with SEM *in-situ* mechanical testing using a nanoindenter, is capable of characterizing all these features, with a sub-100nm resolution, at successive deformation steps and while the material is under load, making it ideal to study small-scale mechanics. However, HR-EBSD is a near surface technique, where only the first few tens of nm are probed underneath the surface. Surface effects are extremely important in small-scale mechanics and surface characterization might not be representative of what is going on in the material during deformation. 3D HR-EBSD technique has been developed to answer this issue and fully characterize the strains, stresses and crystal defect distributions in the deformed materials. Sub-100nm^3 voxel resolution can be achieved by applying successive FIB-slicing and HR-EBSD mapping. We applied this technique to study fracture mechanics in single crystal tungsten at different scales, strain rates and temperatures. The 3D defect distribution below the crack tip help understanding the brittle-to-ductile transition in this material. The results show that surface effects are more pronounced at low temperature.

A novel microtensile uniaxial set-up, equipped with a laterally compliant silicon gripper has been developed to overcome typical issues in small-scale tensile set-ups, particularly sample fabrication, sample handling and misalignment. Both gripper and specimen geometry has been optimized by FE modelling to replicate stress profiles comparable to macroscopic samples defined by ASTM 638. This system has been developed for *in-situ* HR-EBSD and used to study scale effects in twin initiation and propagation in single crystal magnesium. The results show that plastic deformation, with strong non-Schmid factor dislocation activities in the basal plane, occur before twinning at the micron-scale. Tensile twinning is rapidly followed by double twining mechanisms, with the development of compression twins in the middle of the tensile twin. The scale effect on single and double twins development will be discussed.

TEM IN-SITU DEFORMATION OF MGNESIUM-YITTRIUM ALLOYS

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Key Words: In-situ, TEM, Magnesium, Dislocation.

Yttrium (Y) addition is known to be effective in enhancing the formability of magnesium (Mg) alloys by reducing the plasticity anisotropy of the hexagonal crystal, viz. improving the ease of <c+a> dislocation slip in relation to the <a> dislocation slip. In this work, in-situ TEM compression test has been carried out to observe the nucleation and movement of <c+a> dislocations in two Mg-Y binary alloys with different Y contents (0.4 wt.%Y versus 4 wt.%Y). The results have shown (Figure 1) that in the Mg-0.4Y alloy, the edge segments of <c+a> dislocations are sessile. The increased Y content in the Mg-4Y alloy has shown (Figure 2) to increase the <c+a> dislocation mobility and the cross-slip propensity. The results will be discussed with computer simulation results published.

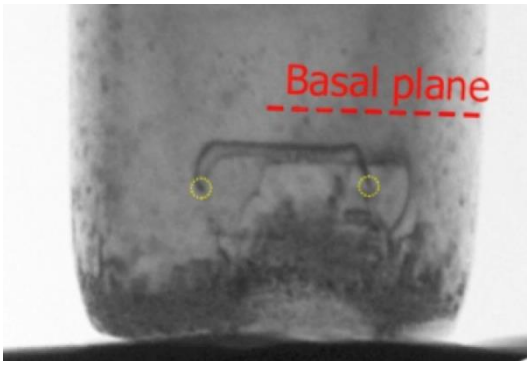

Figure 1 – A snapshot showing <c+a> dislocations in Mg-0.4Y alloy. The long edge segments along the basal plane trace can be observed.

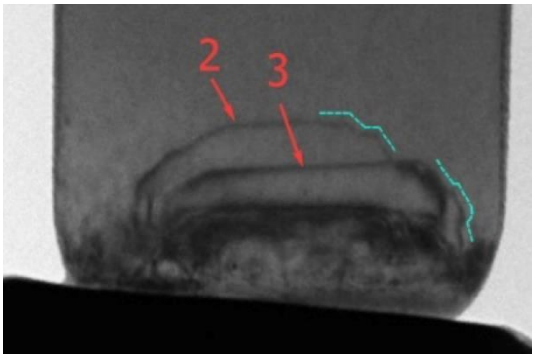

Figure 2 – A snapshot showing <c+a> dislocations in Mg-4Y alloy. The edge segments are less linear decorated with multiple cusps.

IN SITU NANO-INDENTATION OF AU CRYSTALS IMAGED BY BRAGG COHERENT X-RAY DIFFRACTION

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Key Words: *in situ* nano-indentation, Bragg coherent X-ray diffraction imaging, Au crystal, twin

The mechanical properties of micro- and nanostructures were demonstrated to vary significantly from their bulk counterparts. Despite numerous studies, plasticity at the nanoscale is, however, not fully understood yet. *In situ* experiments are perfectly suited for the fundamental understanding of the onset of dislocation nucleation. Recently, we developed a scanning force microscope (SFINX) which is compatible with 3rd generation synchrotron beamlines allowing for *in situ* nano-mechanical tests in combination with nano-focused X-ray diffraction [1] such as coherent X-ray diffraction imaging (CDI). This novel lensless imaging method retrieves the sample scattering function from a coherent X-ray diffraction data set using computational inversion algorithms, thus determining the phase of the scattered amplitude, which is not directly measured by a detector. In Bragg condition, the retrieved phase is directly related to the displacement field and, hence to the strain within a crystal.

Our previous BCDI studies on indented Au crystals demonstrated the capability to imaging a single prismatic loop induced by nano-indentation and trapped inside the crystal [2]. Since any movement of diffractometer motors may induce vibrations that eventually lead to damaging the nano-crystal under load, ordinary rocking scans are not suitable for recording 3D reciprocal space maps *in situ*. Scanning the energy of the incident X-ray beam instead allows for probing the intensity distribution in reciprocal space without any detrimental vibrations [2]. Here, we report about the *in situ* nano-indentation of Au crystals with and without containing a twin boundary parallel to the crystal-substrate interface where the evolution of both strain and defects was imaged by multi-wavelength (mw) BCDI.

Figure 1(a) shows the electron densities for two parts of a twinned Au nanocrystal reconstructed from mw-BCDPs measured at the Au 200 Bragg peaks. The phase, which is directly related to the displacement field inside the structure, is presented in Fig. 1(b) for a gold crystal during nano-indentation allowing for following the evolution of the morphology, the strain field, and dislocations. With increasing applied mechanical load, defects, probably prismatic dislocation loops, appear at about half-height of the indented crystal, which disappear after unloading [4].

To the best of our knowledge, this is the first time that mw-BCDI has been successfully employed during *in situ* experiments providing direct insight into the plasticity at the nanoscale and, in particular, the onset of defect nucleation.

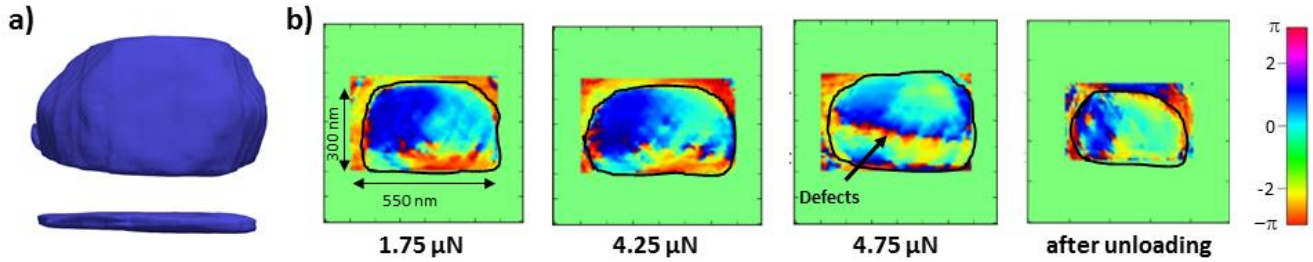

Fig. 2: a) Reconstructions of the upper and the lower part of a twinned Au crystal. b) Z-y cuts of the retrieved phase for an indented Au crystal at various loads as well as after unloading.

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TEM OBSERVATION AND IN SITU COMPRESSION TESTS OF TRANSITION ALUMINA PREPARED BY HIGH PRESSURE COMPACTION AT ROOM TEMPERATURE

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Key Words: alumina; in situ; transmission electron microscopy; plasticity; phase transformation

The behavior of ceramics at the nanometer scale strongly differs from the one of the corresponding bulk material. For instance, strong plastic deformation has recently been reported in isolated nanometer-sized alumina nanoparticles or MgO nanocubes, when tested *in situ* in a transmission electron microscope (TEM). This plastic behavior may also occur in a powder during the compaction process, even at room temperature. Controlling plastic deformation of nanoparticles during the ceramics processing might be a way to enhance their properties or to improve the processing route (compaction and sintering steps, for instance). We present here a comprehensive study of the mechanical behavior of transition alumina in the compacted powder.

Transition alumina nanoparticles have been compacted at room temperature under different uniaxial pressures (5 GPa, 15 GPa and 20 GPa) in a diamond anvil cell or in a Paris-Edimbourg press. Thin foils of these compacted powders can be prepared by Focused Ion Beam machining (FIB) and analysed by TEM (figure 1a). High ResolutionTEM observations and diffraction patterns analyses unambiguously revealed that nanoparticles underwent plastic deformation in the compacted powder. A study of these HRTEM images coupled with Fast Fourier transforms to get the associated diffraction patterns show that the deformation involves the {110} lattice planes, and the slip system {111} <110>. These observations are in agreement with the deformation observed on a single nanoparticle during an in situ nanocompression test inside a TEM. Moreover at high pressure, phase transformation can be evidenced.

To go further on the understanding of mechanisms that may occur during compression of a powder, thin foils have been prepared from the compacted powder and tested in situ in a TEM (Figure 1b). Several imaging conditions have been investigated to follow the nanoparticle movement and/or their deformation during the compression. First results obtained on the compression of thin foils will be presented and discussed.

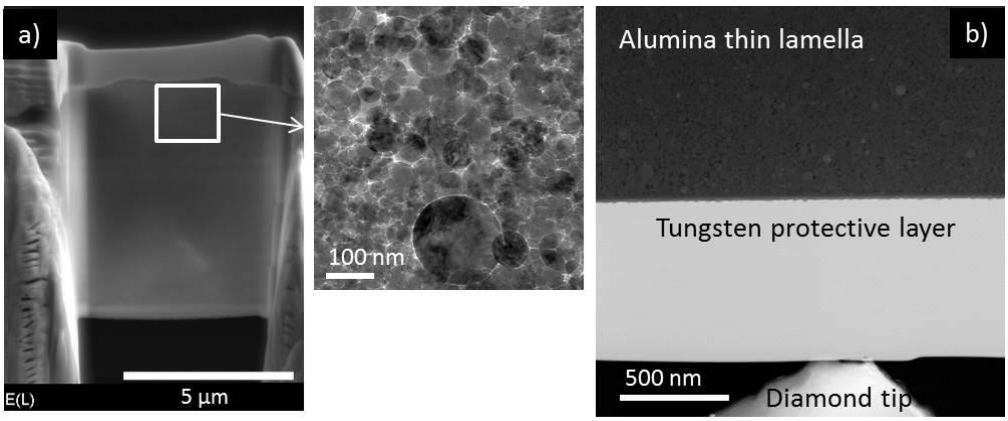

Figure 3 – a) alumina thin lamella prepared by FIB milling, b) alumina thin lamella during in situ nanocompression experiment in TEM observed in STEM mode

DETERMINATION OF PRECIPITATE STRENGTHENING IN Al-Cu ALLOYS THROUGH MICROPILLAR COMPRESSION: EXPERIMENTS AND MULTISCALE SIMULATIONS

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Key Words: Precipitate strengthening, micropillar compression, dislocation dynamics, crystal plasticity.

Al-Cu alloys are efficiently strengthened by different types of precipitates: Guinier-Preston zones, θ'' (Al_3Cu) and θ' (Al_2Cu). The contribution of each type of precipitate to the strengthening of the alloy was determined by means of a high-throughput strategy based on micropillar compression. To this end, an Al-4 wt.% Cu alloy was manufactured by casting, following by several homogenization heat treatments at high temperature. The alloy was aged at 23ºC and 180ºC for different times to produce different precipitate structures [1]. Micropillars were machined using a focus ion beam in grains oriented for single and multiple slip and compressed at ambient temperature. The critical resolved shear stress was determined as a function of the applied strain for micropillars with different sizes oriented for single slip to assess the size effect. It was found that the properties of the bulk crystals could be obtained by testing square micropillars with cross-section > 5 x 5 μm^2. In addition, the precipitate type and spatial distribution as well as the mechanisms of dislocation/precipitate interaction were studied in the transmission electron microscope from lamella extracted from the deformed micropillars. It was found that Guinier-Preston zones and small θ'' precipitates (< 50 nm) were sheared by dislocations while dislocations formed Orowan loops around large θ' precipitates. Afterwards, the effect of latent hardening for the different types of precipitates was studied by compression of micropillars oriented for double slip (coplanar and non-coplanar) as well as for multiple slip.

In parallel, the critical resolved shear stress in the overaged Al-Cu alloys containing large θ' precipitates was simulated by means of dislocation dynamics simulations using the discrete-continuous method in combination with a fast Fourier transform solver to compute the mechanical fields [2]. Simulations took into account the effect of precipitate shape, orientation and volume fraction as well the elastic mismatch between the matrix and the precipitate, the stress-free transformation strain around the precipitate and the dislocation character as well as dislocation cross-slip.

In addition, the results of the micropillar compression tests were used to calibrate the latent hardening parameters of a crystal plasticity model, so they can be used to predict the mechanical behavior of polycrystals by means of computational homogenization. Overall, the results of this investigation show how micropillar compression can be used as a high-throughput technique to obtain the bulk properties of precipitation-strengthened alloys as well as to validate the results of simulation strategies at lower length scales (dislocation dynamics) and to provide input information for simulations at larger length scales (computational homogenization of polycrystals).

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EFFECT OF SAMPLE SIZE AND GRAIN BOUNDARIES ON DISLOCATION STRUCTURES AND DAMAGE EVOLUTION IN SMALL SCALE SAMPLES: A MICRO-FATIGUE INVESTIGATION

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Key Words: Micro-Fatigue, cell structures, single and bi-crystals.

Fatigue failure, one of the most investigated failure mechanisms, occur in a wide variety of engineering components. The recent growing interest in small scale materials for sensors or actuators have raised new questions regarding the length scale influence on fatigue. As the scale is in the range of typical fatigue dislocation structures, size limitations on the formation of persistent slip bands (PSBs), cell structures and the initiation of fatigue cracks at grain boundaries (GB) are some of the relevant questions to be answer.

Grain boundaries are important planar defects providing substantial strengthening mechanisms in polycrystalline materials. However, due to elastic and plastic incompatibilities additional stresses are created at grain boundaries, which could lead to slip transfer, fatigue crack nucleation, etc. Micron-sized bi-crystals can be employed to estimate local stresses and strains, which are associated with the stress-strain response, to get a better understanding of the role of GB. *In situ* micro-fatigue experiments not only provides information of microstructure and damage evolution, but also of local stresses (Figure 1). On that account, our research focuses on cyclic fatigue of single and bi-crystalline micro-samples in order to study the influence of the sample size, crystal orientation and grain boundaries on damage morphology and dislocation structures.

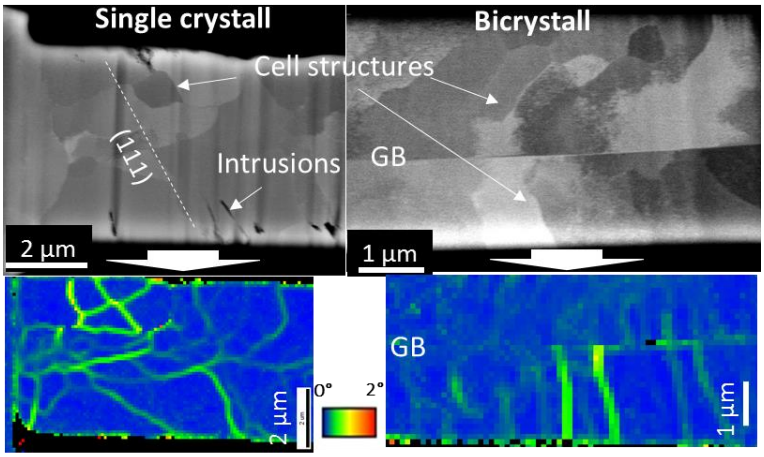

Figure 1. Fatigue damage and dislocation structures in a single and bi-crystalline micro-sample.

THE INFLUENCE OF 3-D INTERFACIAL STRUCTURE AND MORPHOLOGY ON THE MECHANICAL BEHAVIOR OF NANOCOMPOSITES

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Key Words: Ductility, strength, toughness, multilayers, mesoporous

2-dimensional (2D) sharp interfaces with distinct boundaries demarcating an abrupt discontinuity in material properties in nanolayered composites have been studied for almost twenty years and are responsible for enhanced behaviors such as strength, radiation damage tolerance, and deformability. However, 2-D interfaces have their limitations with respect to deformability and toughness. 3D interfaces are defined as heterophase interfaces that extend out of plane into the two crystals on either side and are chemically, crystallographically, and/or topologically divergent, in three dimensions, from both crystals they join. Here, we present the mechanical behavior of two different classes of nanocomposites: 1.) nanolayered Cu/Nb containing interfaces with 3D character and 2.) Tungsten-based 3D ordered mesoporous composites consisting of a porous W scaffolding with silicon carbide or silicon nitride infill. Micropillar compression results show that the strength of Cu/Nb nanocomposites containing 3D interfaces is significantly greater than those containing 2D interfaces. Shear banding in 3D Cu/Nb is observed during pillar compression with retention of continuous layers across the shear band. We will present our recent results on deformation of such 3-D interfaces and structures, and describe this evolution mechanistically through the use of atomistic simulations.

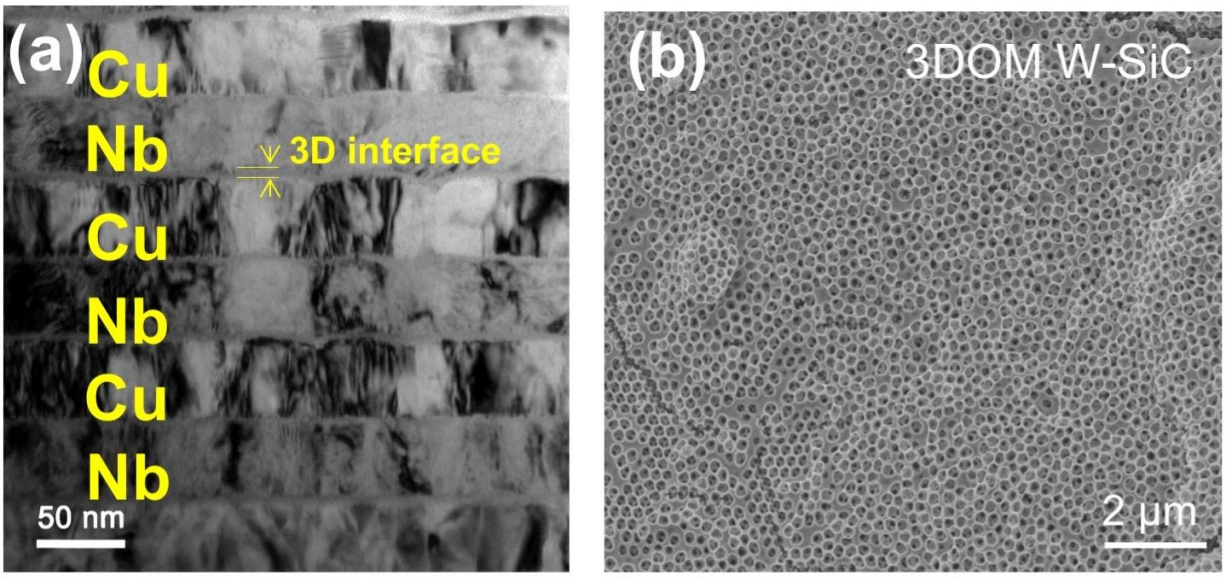

Figure 4 – (a) Bright Field TEM micrograph of Cu-Nb vapor deposited nanocomposite containing 3-D interfaces of chemically intermixed character. (b) SEM micrograph of 3D-ordered mesoporous structure consisting of W ligaments coated with SiC.

ON MICROSTRUCTURAL CONSTRAINTS FOR SLIP TRANSFER IN NANOTWINNED SILVER

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Key Words:Dislocation-grain boundary interactions, coherent twin boundary, slip transmission, nanotwinned fcc.

Micromechanics of bicrystals with selected grain boundary (GB) types is an effective method of understanding dislocation – GB interactions. Micropillar compression tests on bicrystals containing a coherent twin boundary (CTB) [1,2], show that CTBs act as a weak obstacle for slip transfer unlike general high angle grain boundaries. Perfect slip transmission of dislocations through CTBs has been found to be similar to cross slip in fcc metals, and therefore denotes as cross-slip-like slip transfer [2,3].

In the present study, we extend the argument of perfect slip transfer to multiple closely spaced CTBs investigated on nanotwinned Ag films. Nearly 120 micropillars with CTBs parallel to the loading direction and varying twin spacing were investigated. Perfect slip transfer is observed not only across one CTB, but was also observed at several CTBs down to nanoscale spacing. The reduction in twin spacing results in an increase of the yield strength. The study addresses size scaling due to multiple weak constraints for dislocation motion and underlying deformation mechanisms. Effect of coherent, incoherent twin boundaries is discussed under the condition for strain compatibility and in the view of various material parameters such as stacking fault energy and shear modulus.

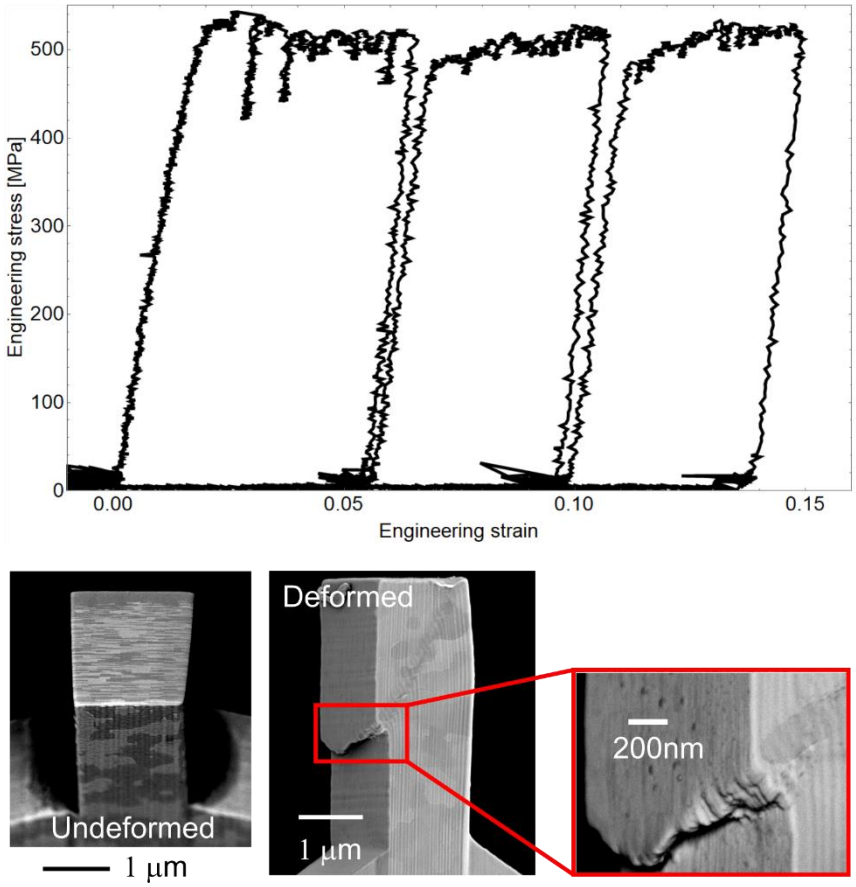

Figure 5 – Typical stress- strain curves and continuity of slip steps in micropillar compression of nanotwinned Ag

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TWIN BOUNDARIES: OBSTACLE FOR OR SOURCES OF DISLOCATIONS

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Key Words: Ductility, Strength, Twin Boundary,

Nanotwinned materials are known for their high strength and ductility, i.e. damage tolerance. The fundamental origin of the damage tolerance is yet not fully clear: While the high strength can be explained by the obstruction of dislocation motion caused by numerous twin boundaries, the origin of the unexpected ductility remains unclear. Classically, damage occurs as soon as the geometric softening due to localization cannot be countered by strain hardening. Since high strength materials typically do not exhibit pronounced strain hardening, slip localization – hence failure – sets in at low strains. But why doesn´t that happen in nanotwinned materials?

To shed light on the damage tolerance we are using nanoindentation with spherical indenters to measure the statistical pop-in behavior of copper single crystals and in the vicinity of coherent □3 twin boundaries (TBs). The pop-in, i.e. a sudden burst in displacement, occurs at the transition from elastic to plastic deformation. We further correlate the pop-in force to the maximum shear stress beneath the indenter, which we interpret as the dislocation source activation stress. Our experiments show that the stress for operating a dislocation source in the vicinity of a TB is substantially lower than the one for dislocation source operation in the grain. Furthermore, the narrow distribution of pop-in stresses near TBs suggest that dislocation nucleation at TBs is omnipresent.

Based on our experiments we propose, that slip localization in nanotwinned materials is suppressed by a unique dislocation multiplication process occurring at imperfections of the otherwise coherent □ 3 TBs. This dislocation multiplication process can only operate few times at a single imperfection, which suppresses slip localization and facilitates damage tolerance.

Metallic glasses (MG) can simultaneously exhibit an enormous strength and fracture toughness – two properties classically rendering a material damage tolerant. However, MG still suffer a brittle-like fracture during tensile loading. The lack of ductility in tension can be related to the nature of shear bands – a subject widely studied over the last decades but still not thoroughly understood.

PROBING GRAIN BOUNDARY RELAXATION IN ULTRA-FINE GRAINED TANTALUM BY MICROMECHANICAL SPECTROSCOPY IN AN SEM

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Keywords: In-situ micromechanical testing; mechanical spectroscopy; ultrafine-grained Tantalum

The study of grain boundaries (GBs) in polycrystalline materials is a field of major interest, since many physical properties, such as thermal and electrical conductivity, magnetic coercitivity, strength or fracture toughness, are influenced by the actual structure of GBs. One of the main challenges in investigating them is the fact that techniques capable to resolve their structure, for example transmission electron microscopy, require very small sample volumes. However, the necessary removal of the surrounding material might change the natural state of the GB by elimination of surrounding material constraints. To counteract this influence, one could apply indirect measurements such as internal friction to probe changes in the GB structure. However, given the ongoing trend towards miniaturization and integration, most of these macroscopic techniques are at their limit.

In our current work, we developed a miniaturized technique for performing mechanical spectroscopy based on micronized bending beams in conjunction with a nanoindenter equipped with a continuous stiffness measurement module in-situ in a scanning electron microscope (SEM). We apply this miniaturized spectroscopy technique to study grain boundary relaxations of ultra-fine grained tantalum micro bending beams in-situ in the SEM, where we assess the influence of a thermal relaxation treatment on the GB structure.

GRAIN-SCALE INVESTIGATION OF THE ANISOTROPY OF PLC-TYPE PLASTIC INSTABILITY

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Keywords: Portevin Le-Chatelier effect, anisotropy, magnesium, nanoindentation, micromechanisms

Various aspects of Portevin Le-Chatelier (PLC) type plastic instability, particularly the influence of strain rate, temperature and precipitation on the phenomenon have been investigated. Such investigations give insights into the underlying governing mechanisms and provide the basis for developing mechanistic and numerical models for these mechanisms. One aspect that is yet to be understood is the influence of anisotropy on plastic instability. So far, experimental efforts aimed at understanding this influence have been focussed on the influence of sample orientation and texture. However, direct measurement of the response of single crystals during uniaxial testing is essential for accurate characterization of the influence of anisotropy. Yet, such an endeavour is largely limited by the difficulty of producing single crystals of technical alloys. Insight into the orientation dependence of plastic instability is thus achieved in this work with a combination of spherical nanoindentation of single grains of Mg AZ91 and local orientation image analysis of cross sections of the nanoindents. Our results indicate that the local stresses arising from the underlying mechanisms that govern plastic instability in this alloy are strongly orientation dependent. In this talk, we will highlight the origin of the orientation dependence and the influence of twinning, and discuss the implications for macroscopic deformation.

NANO-MECHANICAL BEHAVIOR OF BCC IRONS CHARACTERIZED THROUGH NANOINDENTATION AND TEM IN-SITU STRAINING

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Key Words: Dislocation, Dynamic motion, BCC irons, TEM in-situ straining, Grain boundary

Local mechanical behaviors were investigated through nanoindentation and TEM in-situ straining techniques for bcc irons. Pop-in phenomenon that corresponds presumably to local plasticity initiation was detected on load-displacement curves with major parameters of critical load *Pc* and corresponding excursion depth *Dh* [1]. The *Pc* decreases significantly when indent are made on a grain boundary, which indicates a role of dislocation source of a grain boundary. Alloying elements including interstitial or substitutional atoms have an effect on increasing *Pc*. Since the maximum shear stress underneath the indenter is estimated in an order of a theoretical strength, the event can be understood as dislocation nucleation from defect-free region in a crystal. Dislocation structures underneath the indenter were observed through TEM before and after a pop-in event [2]. No dislocations were observed before initiation while considerable dislocations were generated right after the event. These results suggest that dislocation nucleation and multiplication occur drastically upon plasticity initiation based on collective dislocation motion. In-situ TEM compression tests were performed for interstitial-free (IF) steel to get the relationship between an evolution of dislocation structure and flow stress [3]. Figure 1 shows stress-strain curves (a) and TEM images (b, c) of dislocation structures of the sample. The initial dislocation density was quite low, then after the yielding, the dislocation density increased gradually with strain and the corresponding flow stress decreased edgingly, indicating a significant strain softening. The flow stress was plotted as a function of the dislocation density and the stress exponent *m* was evaluated based on the combination of Johnston and Gilman model and Orowan model. The *m* value is lower than that in the case of edge dislocation dominant condition that is previously shown in literatures. This is reasonable because the stress exponent *m* goes down with the much lower mobility of screw dislocation than that of the edge dislocation in bcc structure.

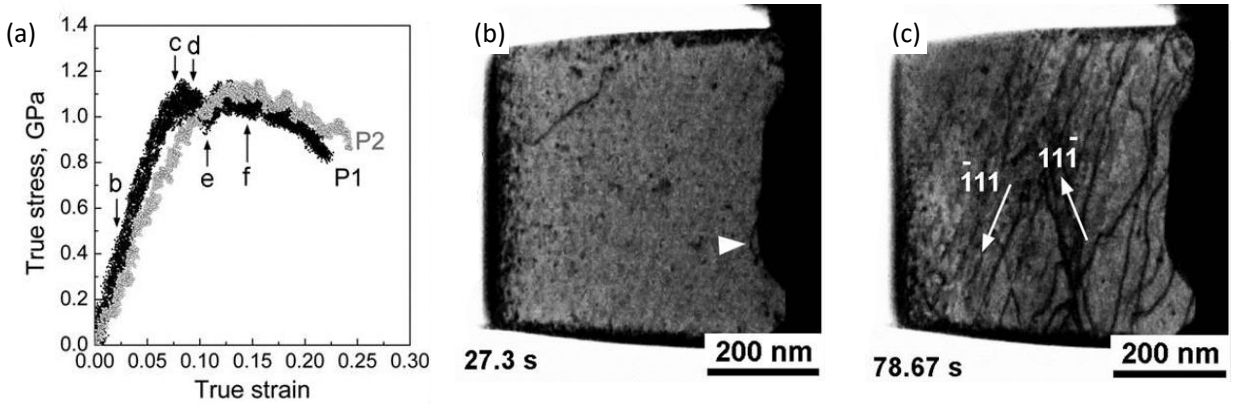

Figure 6 – (a): Stress-strain curves of IF steel obtained through micro-compression test in a TEM. TEM images of the sample before (b) and after (c) yielding showing an evolution of screw dislocations during deformation. The sample showed remarkable strain softening with increasing screw dislocation density.

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NANOMECHANICALTESTING OF BCC MICROPILLARS:
POWER LAWS AND LATTICE RESISTANCE CORRELATIONS

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Key Words: Micropillar Compression, BCC metals, Friction Stress

It is now well established that the compression plastic flow stress, σ_p, of metallic micropillars increases with decreasing sample diameter. With fcc metals, if the pillar flow stress and pillar diameter are normalised by the shear modulus resolved onto the active slip system, μ, and Burgers' vector, b, respectively, the data follows an empirical relation of

$$\frac{\sigma_p}{\mu} = A\left(\frac{d}{b}\right)^n \quad (1)$$

with a "universal exponent" of approximately n = -0.67.

Specimens from bcc metals tested in microcompression show significant differences in their behaviour with size exponents showing a range from -0.2 to -0.8. It is generally believed that the difference in the behaviour of bcc and fcc metals is related to the behaviour of dislocations in the different lattices. In fcc materials dislocations are highly mobile, show similar mobility for edge and screw character and show little need for thermal activation. However, in bcc materials the dislocation core structure of a screw dislocation leads to a significantly higher Peierls' stress and lower mobility compared to edge dislocations, requiring a degree of thermal activation. In which case a different relation may be more appropriate with:

$$\sigma_p - \sigma_b = A' d^{n'} \quad (2)$$

Where, σ_b, is the temperature dependent lattice friction stress that must be overcome to allow plastic flow.

Here we will show that if the data is plotted in the form of equation (1), if equation (2) is valid there should be a strong correlation between the slope of the log(σ)/log(d) plot (n in equation 1) and the proportionality materials constant A. This prediction is shown to be consistent with micropillar compression experiments carried out on a range of bcc metals over a range of testing temperatures in this study (figure 1) and to also hold true for data in the published literature. Further analysis of the data can be used to predict a "natural power law" for micropillar compression, n' in equation 2, which for the case of bcc materials is found to be close to the empirical value of -0.67 determined for fcc materials.

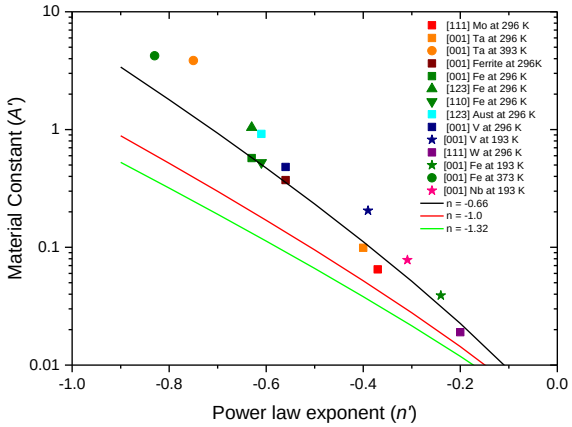

Figure 7 – Correlation between the power law exponent, n, and the materials pre-exponent constant, A, for a number of bcc metals

SIZE EFFECT IN POLYMER-SUPPORTED ULTRATHIN METALLIC GLASS FILMS

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Key Words: thin film metallic glass; polymer substrate; tensile; size effect; shear band.

Although metallic glasses (MGs) exhibit a unique combination of mechanical and chemical properties, their application as structural or functional materials is hindered by the lack of ductility which leads to catastrophic brittle-like fracture. When the size of a MG sample is reduced below some critical value, typically of the order of a few hundred nanometers, then considerable ductility can be observed. However, this size effect was demonstrated so far mostly by nanomechanical testing inside a transmission electron microscope using samples prepared by focused ion beam (FIB) milling. Whether the ductile-like behavior of submicrometer-sized metallic glasses is a real “intrinsic” size effect or it is rather caused by extrinsic factors like sample shape, ion beam effect or parameters of the testing setup is currently a subject of extensive discussions in the community. In this contribution the tensile properties of thin film $Pd_{82}Si_{18}$ MGs grown by sputter deposition on a polymer substrate are considered. The integrity of the MG films during stretching was monitored by in-situ measurements of the electrical resistance. An overview of electro-mechanical behavior of considered films is demonstrated in Fig. 1.The 250, 100, and 60 nm thick films fail in a brittle manner at 2% strain through propagation of long cracks perpendicularly to the straining direction. The rapid crack propagation in these films results in rapid increase of in-situ resistance signal. The size effect on the deformation behavior appears when the film thickness drops below 15 nm. The 7 nm thick films with the same composition show a crack-free deformation up to a strain of 7%. Even at higher strains no brittle-like failure but rather short and isolated cracks are observed. Cyclic tensile loading revealed extreme fracture resistance of ultrathin amorphous films showing no cracks after 30000 stretching cycles with a strain amplitude of 3%. Since all tests are performed at ambient conditions on films deposited using an industrially scalable process, the demonstrated size effect can be directly utilized for applications, such as protective coatings, nanoelectromechanical devices or half-transparent conductive layers for flexible electronics.

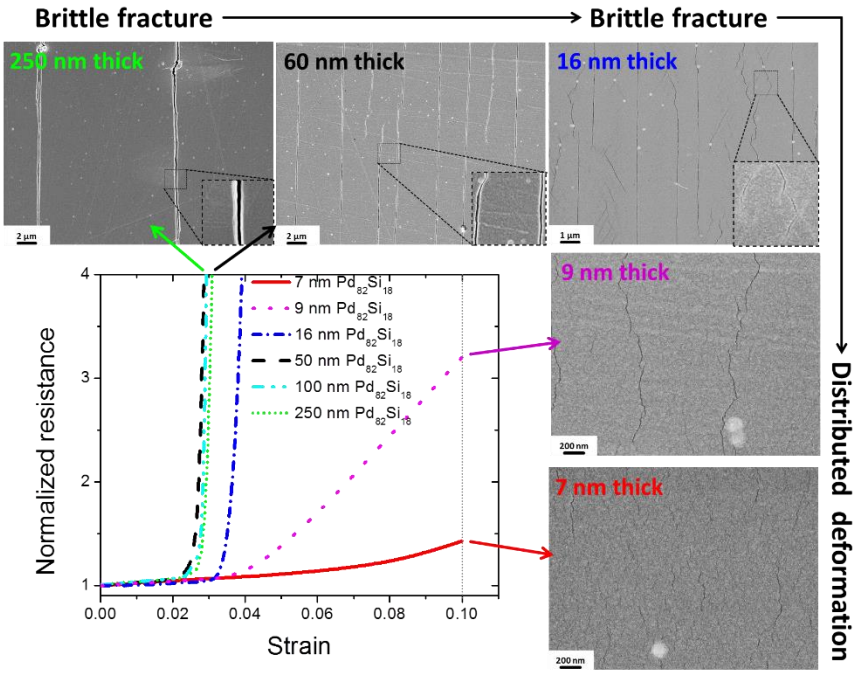

Fig. 1. Overview of electro-mechanical behavior of thin metallic glass films on polymer substrate.

SUPPRESSING DAMAGE IN DUAL PHASE STEEL: INSIGHTS FROM MICROMECHANCIS

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Key Words: BCC ferrite, slip planes, damage nucleation.

Single crystalline ferrite and martensite islands were extracted from two different dual phase (DP) steel grades by focused ion beam milling (FIB) and tested by in situ pillar compression. Three slip plane families {110}, {112}, {123} in bcc ferrite are all observed to be active and their corresponding mean critical resolved shear stress (CRSS) of 3µm pillars are found to be nearly identical, i.e. 147 ± 6, 143 ± 9, 146 ± 4 MPa. Non Schmid contributions either due to tension-compression anisotropy or a size dependent breakdown of Schmid's law plays a minor role in our case. Martensite pillars contain several interfaces which makes them deform isotropically and without distinct slip traces. The pillars exhibit a high mean compressive yield strength up to 2880±49 MPa and a low scatter of 188±17 MPa. By comparing two DP steels possessing identical ultimate tensile strength, the sample with softer ferrite phase and a harder martensite yields at a lower stress and shows a larger fracture elongation. The post mortem investigation of the macroscopic sample indicates that a larger mechanical heterogeneity between ferrite and martensite increases the amount of crack initiation sites but the improved local strain hardening capability imposed by a softer ferrite matrix suppresses catastrophic failure considerably.

We thank Gerhard Dehm (MPIE) for discussions and the DFG for financial support via TRR188 project.

COMPRESSION OF GOLD SUB-MICRON CRYSTALLITES: METHOD AND EXPERIMENTS

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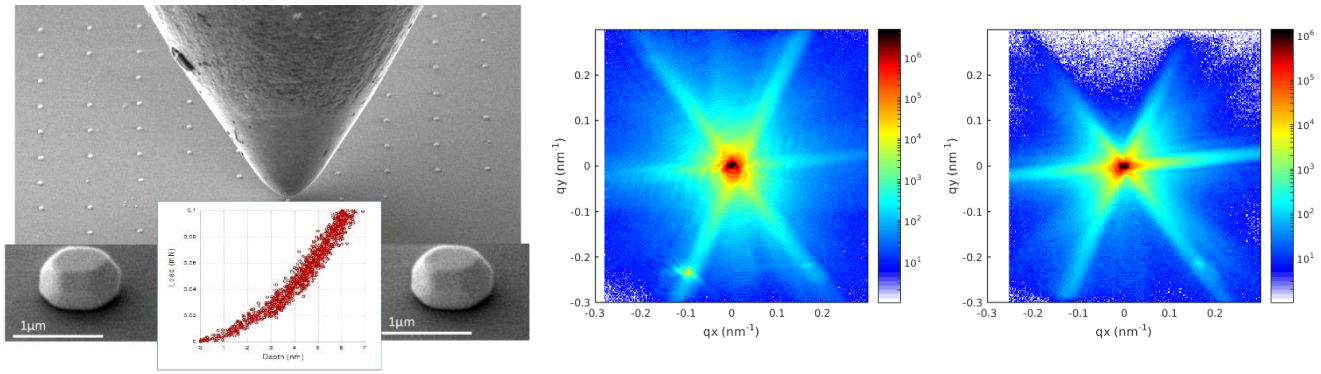
Key Words: Crystalline plastic deformation, stochastic yield, insitu SEM nanomechanical testing, Coherent X-Ray imaging

Understanding and characterizing the mechanical response of individual nanostructure is of great importance for both fundamental prospects and device reliability. Higher flow stress with decreasing sample size is observed together with jerky flow. Compression of pristine submicron gold crystallites yield at very large stress in a stochastic manner, followed by large displacement bursts reaching up to 50% of the initial height [1,2].
In this work, by collecting a large set of measurements, we investigate the small and large strain behavior of crystallites loaded in compression. Large arrays of [111] oriented gold crystallites are prepared by solid state dewetting of initial cylinders of different volumes on sapphire substrates. Dedicated flat punch compression *in-situ* a FEG-SEM (figure 1a) has been carried out in load controlled mode [3]. Microstructure of defects is investigated using synchrotron radiation by nanoscale 3D imaging (Bragg Coherent X-ray Diffraction Imaging) [4] and Atomic Force Microscopy observations. The analysis of the plastic instability and its amount of deformation is carried out taking into account the inertial effect of the instrument, using a 1D dynamic model and Finite Element Method calculations. Simulations are made with different estimates of the shape of each individual crystallite, from an ideal cylinder of equivalent volume to the one based on SEM or AFM observations. We show that prior to the displacement burst, plastic events take place and that the sudden displacement does not necessarily relates to the onset of dislocation nucleation (figure 1b). Moreover, using the collection of measurements, we show that a unique stress-strain response can be obtained which can be used as a lower bound estimate of the mechanical response in compression of the crystallites.



(a) *(b)*

Figure 8 – (a) Flat punch compression of Au crystallite array insitu a SEM-FEG, inset showing a reversible force-displacement, (b) associated coherent diffraction [111] Bragg before and after 'reversible' loading

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DIRECT OBSERVATION OF DISLOCATION PLASTICITY IN FeCrCoMnNi HIGH-ENTROPY ALLOYS

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Key Words: High-entropy alloys, Dislocation plasticity, *in situ* TEM, Strengthening mechanisms, Friction stress

In the past decade, high-entropy alloys (HEAs) have been intensively investigated not only because of fundamental scientific interests, but also their outstanding mechanical properties, for example, high ductility and fracture toughness. Among hundreds of different combinations of principal elements, the equiatomic FeCrCoMnNi alloy, the so-called Cantor alloy, has been studied as a model system, which is a single phase material with face-centered cubic (FCC) structure at room temperature and shows outstanding ductility and strain hardening especially at cryogenic temperatures.

However, dislocation-based deformation mechanisms of HEAs remain elusive and require a fundamental understanding in order to tailor their mechanical properties. Several models have been suggested possible strengthening mechanisms of HEAs, for instance, the high entropy effect and the lattice distortion effect. In the case of the Cantor alloy, the main strengthening mechanism was identified as deformation twinning with critical twinning stress of 720 MPa. At room temperature, dislocation slip by full dislocations is dominant, however, at strains exceeding 20 % and high flow stresses, deformation twinning was also observed. To reveal the hardening mechanism in more detail, direct observation of dislocation plasticity and deformation dynamics is required.

Here, we present a study correlating the microstructure and dislocation plasticity of a single crystalline FeCrCoMnNi FCC single phase HEA by employing in-situ transmission electron microscope (TEM) compression and tensile deformation. Moreover, an atomic-scale chemical analysis is conducted by aberration-corrected scanning TEM energy dispersive X-ray spectroscopy (STEM-EDS) and atom probe tomography to investigate chemical inhomogeneity, for example, precipitate formation or local inhomogeneity.

Compression tests with sub-micron pillars with 250 and 120 nm diameter show less pronounced mechanical size effects in the alloy compared to other FCC metals as the size exponent is measured as 0.53. It suggests that relatively strong inherent hardening processes are present which attenuate the FCC reported size scaling exponent, which is typically 0.6 to 1.0 for pure FCC metals. The elemental distribution and lattice strains at the atomic scale are rather uniform without long-range ordering analyzed by high-resolution scanning TEM (STEM) and atom probe tomography. Finally, dislocation glide motion was directly observed during *in situ* TEM tensile tests. The local shear stress measured from gliding of individual dislocations is exceeding 400 MPa. Kink-pair-like glide behavior and periodic fluctuation in the stacking fault width suggest that local pinning points, severe lattice distortion or short-range ordering hinder dislocation motion in HEAs.

DISLOCATIONS IN LAVES PHASES – PHANTASTICAL BEASTS AND HOW TO UNDERSTAND THEM

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Key Words: Dislocations, Intermetallics, Slip System, Nanomechanics, TEM

How macroscopically hard and brittle materials deform is not well understood in many cases with not even the dominant slip systems known and no critical stresses or dislocation mechanisms available. This is true even for the most abundant type of intermetallic phase, the Laves phases. However, this knowledge is essential to improve many metallic-intermetallic composite alloys, such as Mg with an interconnected Laves network in Mg-Al-Ca alloys preventing creep.

Here, a study combining nanomechanical testing, TEM and atomistic as well as ab-initio simulations will be presented with the Mg_2Ca phase as a model Laves phase. Using indentation of suitably oriented grains, slip trace analysis and TEM at room and elevated temperature, we elucidate the dominant slip systems and their temperature dependence. A high retained strength is found even at 200 °C, which is consistent with the improved creep resistance of Mg-Al-Ca alloys with the Mg_2Ca phase at these high temperatures.

Although the most easily activated slip system in this phase is not the basal plane, it is only this plane for which a dislocation mechanism has been suggested in the form of synchroshear. How this mechanism may be modelled and which configurations of the dislocation core determine the required stress has been unraveled by atomistic modelling for the first time. Finally, we are expanding this work towards the non-basal mechanisms theoretically and towards off-stoichiometric phases experimentally and will briefly review the challenges encountered in attempting both in such complex, ordered crystals.

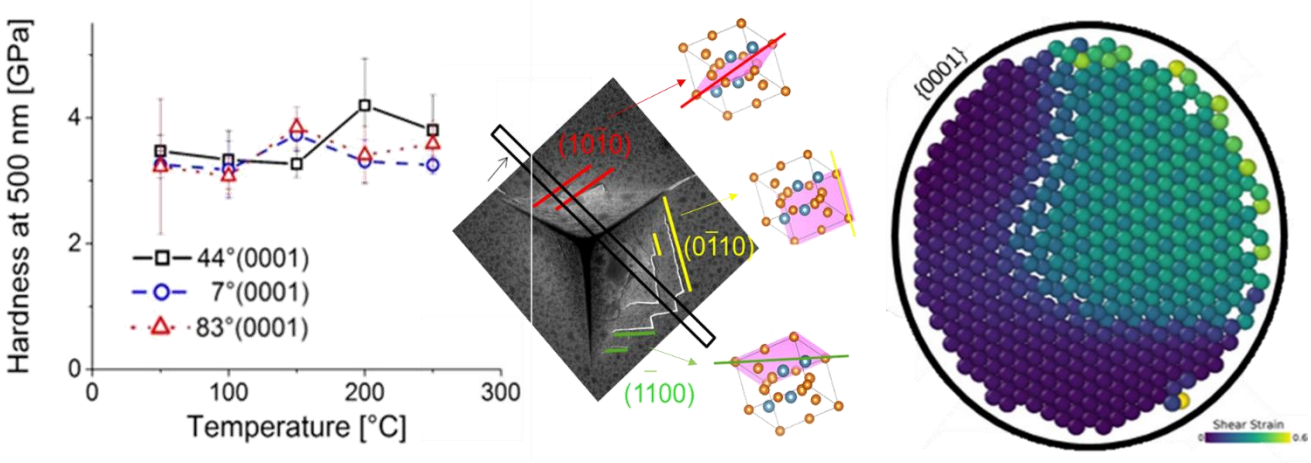

Figure 9 – Nanomechanical testing and atomistic simulation of the slip mechanisms in the Mg_2Ca Laves phase (experiments: unpublished, simulations:[1].)

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NANOMECHANICAL TESTING STUDY OF THE ELEMENTARY DEFORMATION MECHANISMS IN THE Ti_2AlN AND Cr_2AlC MAX PHASES

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Key Words: MAX phases, nanoindentation, dislocations, plasticity.

Abstract: Deformation mechanisms in MAX phases are still not well understood. The complex mechanical behavior of these materials, including mechanical hysteresis, arises both from their crystallography, with a nanolayered structure alternating nitride or carbide layers with metal atoms layers, and from their macroscopic polycrystalline structure, composed of platelets-like grains. In order to distinguish from these two contributions, we focused our study at the sub-micrometer scale, in order to probe the mechanical response of individual grains.

For this purpose, nanoindentation tests were performed at room temperature and at high temperature in single grains of Ti_2AlN samples. The deformation microstructures were then investigated by Atomic Force Microscopy (AFM) through the observation of the slip lines left at the surface by the dislocations, and by Transmission Electron Microscopy (TEM) in cross section through the nanoindents. An automated mapping of crystallographic orientations was also performed using the ACOM (Automatic Crystal Orientation and phase Mapping) ASTAR technique (cf. figure 1). These experiments revealed the presence of highly disoriented domains below the indents, as well as more conventional low angle tilt boundaries associated with dislocation walls. Different possible deformation mechanisms are discussed in light of these observations. In a second approach, spherical nanoindentation tests have been performed in Cr_2AlC single crystals recently synthesized by high temperature solution growth. These samples are thin platelets of few mm^2, oriented along the basal plane. By embedding these platelets in a given orientation (cf. figure 2), it is possible to choose the crystallographic orientation of the indented surface in order to probe the plastic deformation mechanisms in specific configurations. The dislocation structures and the highly disoriented domains have been observed around a same indent by AFM and TEM, and the deformation mechanisms will be discussed in view of these observations.

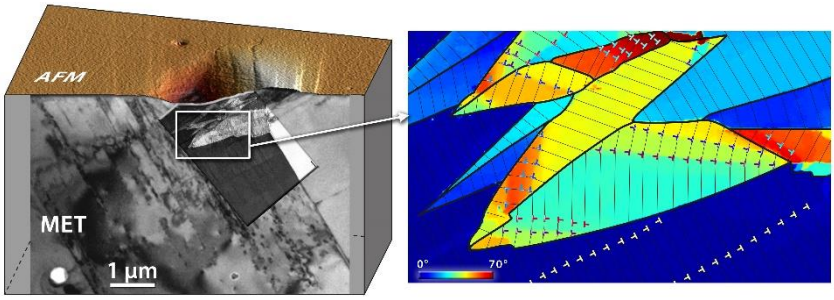

Figure 10 – AFM and cross section TEM analysis of the deformation structure around a nanoindentation imprint performed at 800°C (left side) and local crystallographic disorientations in the same area (right side).

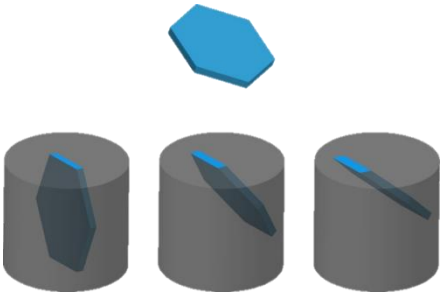

Figure 2 – Cr_2AlC single crystal platelets embedded in a chosen orientation for nanoindentation test in cross section.

SUPERELASITICTY OF $ThCr_2Si_2$-STRUCTURED INTERMETALLIC COMPOUNDS AT THE MICROMETER SCALE

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Key Words: Superelasticity, Microcompression, Density Functional Theory, Single Crystal Growth

The work represents a report of the discovery of superelasticity in $ThCr_2Si_2$-structured novel intermetallic compound ($CaFe_2As_2$) and its hybrid structure ($CaKFe_4As_4$) under "uni-axial" compression at the micrometer scale and discusses the strong possibility of deformation-induced superconductivity switching. They exhibit unprecedentedly large elastic limit (10~17%), ultrahigh strength (3~5 GPa), and repeatable cyclic loading response through the reversible lattice collapse caused by making and breaking atomic bonds.[1-4] This unique superelasticity mechanism produces a modulus of resilience orders of magnitude higher than that of most engineering materials and enables strain engineering, which refers to the modification of material properties through elastic strain. Our experimental and computational results strongly suggest that superconductivity in a high temperature superconductor, $CaKFe_4As_4$, could be turned on/off reliably through this superelasticity process, before fracture occurs, even under "uniaxial" compression. Please note that it is extremely rare to see deformation-induced superconductivity switching under uni-axial deformation, which is the preferred loading mode in engineering applications. Note that our result is only one manifestation of a wider class of such transitions found in over 2500 different $ThCr_2Si_2$-structured intermetallic compounds. If we consider their hybrid structure, there could be a much larger number of similar intermetallic compounds. Therefore, our observation can be extended to search for a large group of superelastic and strain-engineerable functional materials, and, more broadly, will lead to various research opportunities in materials science, solid-state physics, superconducting device engineering, and machine-learning-based materials research.

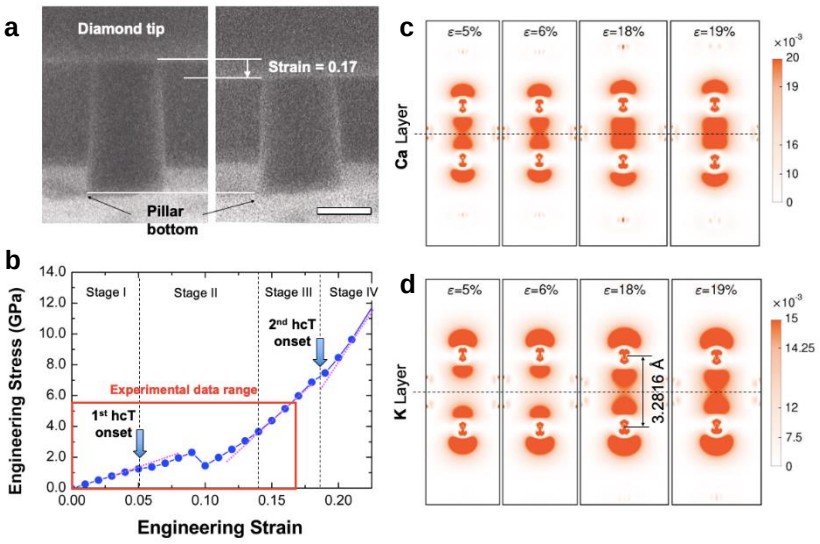

Figure - Superelasticity of $CaKFe_4As_4$. (a) Snapshots of in-situ video right before contact with the diamond tip and right before failure (scale bar, 1μm); (b) DFT simulation results of engineering stress-strain data.; Non-spin-polarized electron density in the ac plane associated with the As-$4p_z$ orbitals near (c) Ca and (d) K at different strains. (c) shows clear bond formation across the Ca-layer by 0.05 strain and (d) shows clear bond formation across K-layer by 0.18 strain.

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SMALL-SCALE MECHANICAL RESPONSE OF CEMENTED CARBIDES: CORRELATION BETWEEN MECHANICAL PROPERTIES AND MICROSTRUCTURE

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Key Words: WC-Co, small-scale, mechanical properties, high speed tests, high temperature tests.

The unique combination of hardness, toughness and wear resistance exhibited by heterogeneous hard materials (e.g. cemented carbides, PCD composites, PcBN systems and generic hard coating/substrate combinations) has made them preeminent material choices for extremely demanding applications, such as metal cutting/forming tools or mining bits, where improved and consistent performance together with high reliability are required. The remarkable mechanical properties of these materials results from a two-fold effectiveness associated with their intrinsic composite character. On the one hand in terms of composite nature: combination of completely different phases (hard, brittle and soft, ductile constituents) with optimal interface properties. On the other hand as related to composite assemblage: two interpenetrating-phase networks where toughening is optimized through different mechanisms depending on the relatively different chemical nature among them.

In particular, this presentation is focused on WC-Co hardmetals, as reference hard material. Large number of studies has been reported, mainly focused on the mechanical behavior of this composite. On the other hand, information on the small-scale mechanical response of these materials is rather scarce. This is particularly true regarding experimental data and analysis on the influence of phase nature, crystal orientation (anisotropy) and interfacial adhesion strength on hardness, deformation and/or damage mechanisms. It is clear that knowledge of these issues is crucial not only to improve the performance of hardmetals but also to develop ceramic-metal composites beyond WC-Co systems.

A systematic micro- and nanomechanical study of the mechanical response of several microstructurally different WC-Co grades is presented. In doing so, nanoindentation technique is implemented and corresponding deformation/damage mechanisms are also investigated. In general, five different approaches are followed to accomplish the main goal of this research: (1) assessment of intrinsic hardness values and main deformation mechanisms as a function of crystal orientation for the carbide phase at room temperature (RT) and also at high temperature (from RT to 600 ºC), (2) determination of effective hardness and flow stress of the metallic binder through massive nanoindentation and statistical analysis, (3) evaluation of the Hall-Petch parameters for the WC-Co as a function of a microstructural parameter (mean free path) by using the methodology presented above, (4) correlation of the microstructure with the hardness and elastic modulus map by using high indentation speed tests, and (5) study of the stress-strain response by means of ex/in-situ compression of micropillars.

It is found that WC-Co composites are strongly anisotropic in terms of hardness at the small scale (microstructure), being the WC hardness for the basal plane about 20-30% higher than for the prismatic and pyramidal planes. It implies consideration of carbides with different crystal orientations as distinct phases for statistical analysis of massive nanoindentation data. Implementation of such testing/analysis protocol indicates a flow stress for the constrained Co-based binder of about 2.6-3.5 GPa. By plotting of the experimentally data as a function of the binder mean free path results in a Hall-Petch strengthening relationship.

Finally, the compression of micropillars points out that main deformation mechanisms are located in the metallic binder although close to the strong interface exhibited by these materials.

NANOMECHANICAL BEHAVIOUR OF INDIVIDUAL PHASES AND SIZE EFFECT IN WC-CO BY MEANS OF HIGH TEMPERATURE NANOINDENTATION AND ELECTRON MICROSCOPY: A STUDY FROM AMBIENT TO HIGH TEMPERATURE

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The dependence of the hardness and deformation mechanism of individual phases in WC-Co on microstructural parameters such as grain size and orientation was investigated by nanoindentation and electron microscopy from ambient to high temperature. At room temperature, the binder phase only exhibits a hardness of about 10 GPa, whilst the hardness of WC grains were measured about 29-30 and 37 GPa for the prismatic and basal orientation, respectively. All WC orientations exhibited a similar decrease in hardness as the temperature increased. A broad range of WC prismatic grain areas ($A_{WC\text{-}prismatic}$), from about 2 to 1000 µm^2, were selected and subsequently indented to investigate any size effect. A slight decrease in the hardness of WC prismatic grains ($H_{WC\text{-}prismatic}$) as a function of $A_{WC\text{-}prismatic}$ was observed. Damage mechanisms occurring in WC-Co during nanoindentation were investigated for the different grain orientation at various temperature. The damage was visualised using electron microscopy near the residual indent as well as focused ion beam sectioning across the indent. The three dimensional distribution of plastic deformation across multiple grains in the vicinity of an indent was examined using Electron Back Scattered Diffraction (EBSD) and Electron Channelling Contrast Imaging (ECCI). The ECCI enabled the observation of crystal defects, especially dislocations, in th plastic zone. The dislocation density and spatial distribution in the deformed WC-Co were compared to that of an untested WC-Co to relate the quantity of defects as well as their origin to the state of stress in the material. The collected data represent useful guidance for manufacturer of hardmetals, provides important information underpinning an understanding of the relationship between WC-Co microstructure and mechanical properties, and also highlight the performance of WC-Co at operating temperatures.

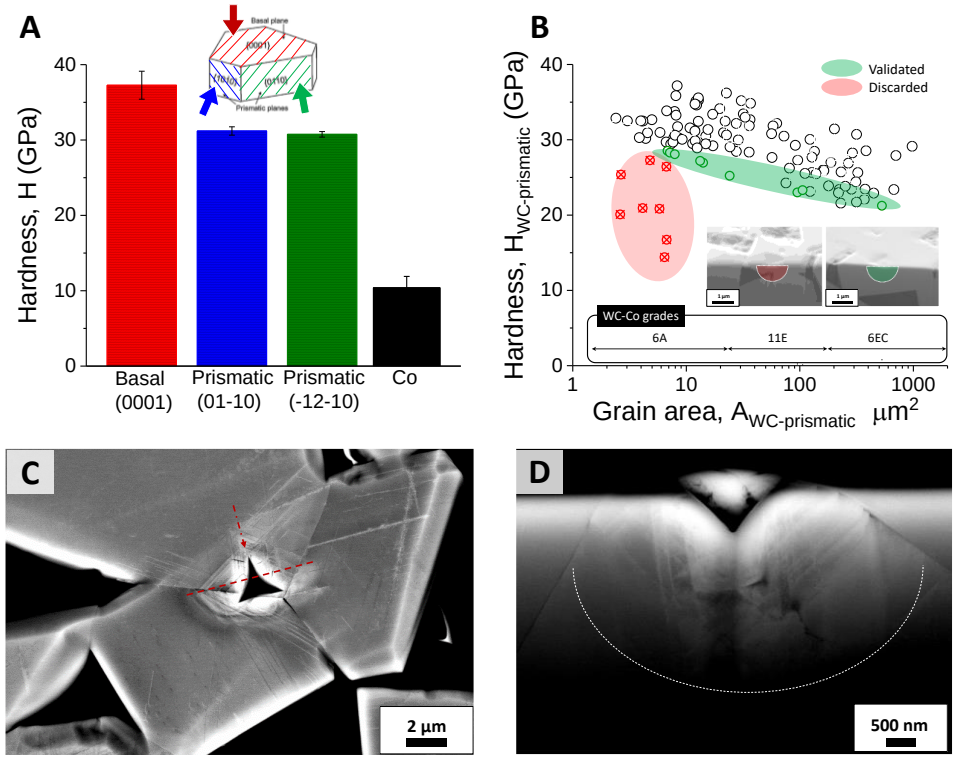

Figure 1. Nanomechanical investigation of WC-Co at room temperature. Hardness, grain size effect, top and cross-section deformation mechanisms evidenced by ECCI (A, B, C and D, respectively).

FRACTURE TOUGHNESS DETERMINATION OF ARC-PVD AND HIPIMS (Al,TI)N HARD COATINGS BY MICRO-CANTILEVER AND PILLAR SPLITTING TESTS

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Key Words: Fracture toughness, micro-cantilever, pillar splitting, in-situ testing, hard coatings

We performed small-scale in-situ fracture experiments in a scanning electron microscope (SEM) using micro-pillar splitting and micro-cantilever bending technique to determine the fracture toughness of five different arc-PVD and HiPIMS (Al,Ti)N hard coatings. All coatings showed brittle fracture with slightly different fracture toughness. In this talk, we discuss on the one hand side the observed differences in the fracture behavior, which are based on the coating microstructure, which depends on the deposition parameters and techniques. Furthermore we want to investigate as to how small-scale fracture techniques can be used as tool to provide necessary information on the lifetime of hard coatings as protection of cutting tools.

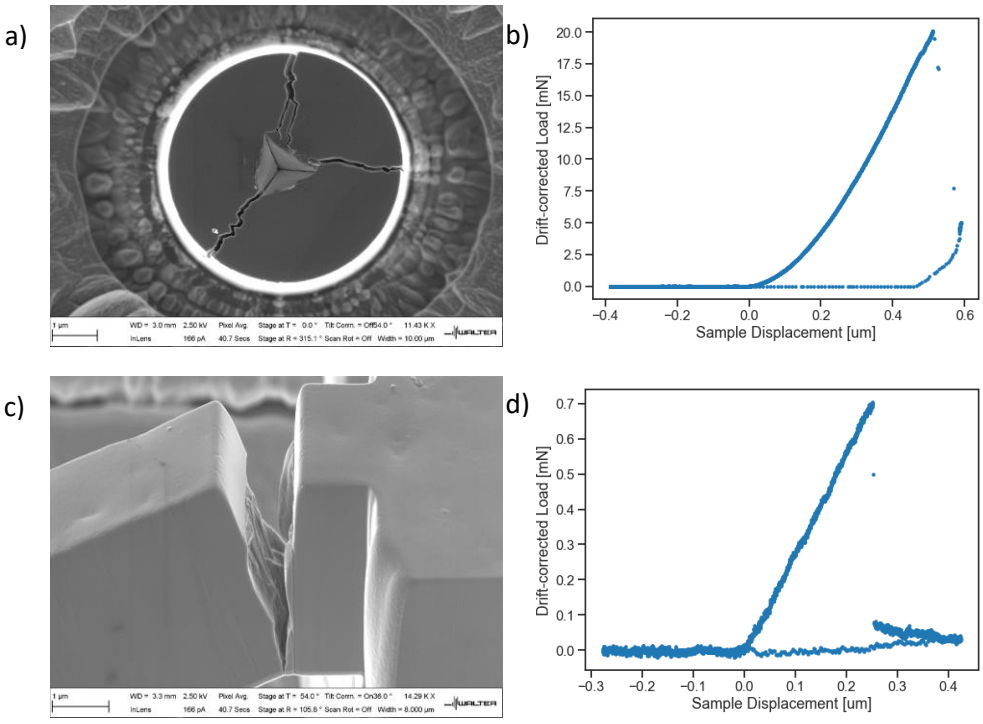

Fig. 1: a) SEM image of splitted micro-pillar b) corresponding force-displacement data, c) SEM image of fractured micro-cantilever and d) corresponding force-displacement data.

THE FRACTURE BEHAVIOR OF Cr_2AlC COATINGS

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Key Words: MAX phase; hard coating; fracture mechanics; micromechanics; Cr_2AlC

The erosion - and self-healing - behavior of Cr2AlC MAX phase coatings has been investigated [1]. It is well known that Cr2AlC coatings can be deposited at temperatures of around 450 °C [2], which is significantly lower than for other MAX phase systems, which often require growth temperatures around 900 °C [3]. To further explore the applicability of the Cr2AlC system in harsh environments, it is necessary to determine its mechanical response. Recent advances in micromechanical testing allow investigating the mechanical properties of hard coatings, especially the fracture behavior, which is of particular interest for several thin film applications. Furthermore, it is possible to deposit the Cr2AlC system with different microstructures, e.g. nanocrystalline or amorphous [2]. Preliminary results revealed a fracture toughness of ~2 MPam1/2 for a coating with columnar morphology. In this investigation, the effect of morphology and microstructure on the fracture toughness of Cr2AlC coatings will be presented.

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STRAIN EVOLUTION AROUND CORROSION PITS UNDER FATIGUE LOADING USING DIGITAL IMAGE CORRELATION

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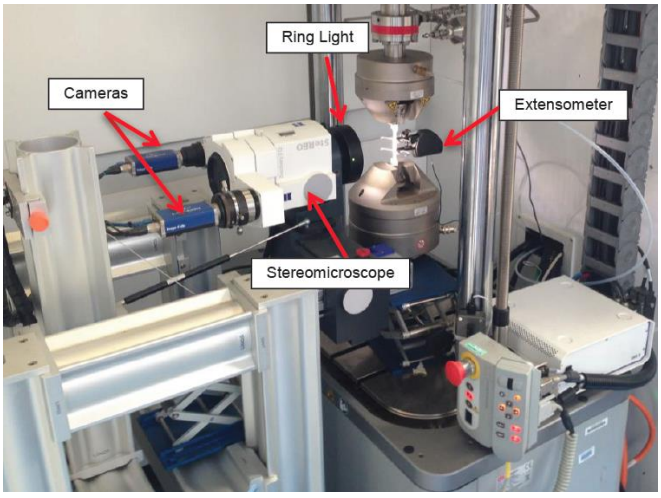
Key Words: Digital Image Correlation, pitting, strain, fatigue

Localised corrosion plays a dominant role in the failure of components and structures exposed to the conjoint effects of corrosion and fatigue through the mechanism of corrosion fatigue. In general materials show good fatigue resistance when tested in air. However, under the synergistic effect of a corrosive environment and cyclic stress, this fatigue resistance is greatly diminished.

Localised corrosion, in the form of pitting, occurs in the early stages of corrosion fatigue. The corrosion pits that initiate and grow in the walls of components and structures can act as precursors to cracking due to a stress concentration around the pits. The pit-crack transition stage of the corrosion fatigue damage process occurs once the corrosion pit is of a size/geometry where it creates a sufficient stress concentration to allow generation of localised strain, which in turn leads to the initiation of a crack. Therefore, it is of interest to determine the effect of pit geometry on the fatigue resistance of materials.

In this study, single and multiple corrosion pits were created using a micro-electrochemical cell, which allowed pit geometry to be controlled. This method enables the effect of a single or multiple corrosion pits on fatigue lifetime to be studied.

In-situ Digital Image Correlation (DIC) was performed on pre-pitted fatigue specimens to examine the evolution of strain around corrosion pits across a range of pit aspect ratios. The aim of the study was to establish whether there is a critical strain concentration at which a crack initiates from a corrosion pit, and whether this critical strain value is dependent on pit geometry and pit-pit spatial relationship. Following single pit studies, two pits were generated on fatigue specimens using different pit aspect ratio and separation distance combinations. DIC was then used to investigate the interaction between neighbouring pits, and its effect on surface strain evolution during crack initiation and subsequent crack coalescence.



DIC Experimental set up

The localised strain data generated from this series of experiments will be used in the validation of the cellular automata finite element modelling approach being developed to develop a unified corrosion fatigue predictive model.

MECHANICAL CHARACTERIZATION OF A TRIBOLAYER CREATED BY HIGH TEMPERATURE FRETTING WEAR IN A CERAMIC/METAL ALLOY CONTACT

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Key Words: glaze layer, high temperature, microcompression, nanoindentation, fretting.

In aeronautics, the blade disk contact, between ceramic and Haynes 25 (cobalt-based alloy) surfaces, is submitted to fretting oscillations, at high temperature, the fretting being an oscillatory movement (at the micrometer order) between two surfaces in contact. This contact has been modeled in the laboratory, showing high friction and wear at temperatures lower than 500°C whereas a sudden decrease of the friction coefficient, and negligible wear is observed above this threshold temperature. The cause of high friction and wear at low temperature was explained in previous paper [1]. At temperatures higher than 500°C, low friction and wear are linked to the formation of a third body, named glaze layer, or tribolayer, this layer being created by compacted and sintered debris, and adhering on both parts, the thickness being between 5-20µm. Its structure and chemical composition was studied in a previous paper [2]. However, understanding the mechanical properties responsible of the glaze layer lubricious properties is still a challenge.

In this presentation, the mechanical properties of the Glaze Layer, measured as a function of the temperature, will be presented, and compared to its tribological properties. The HS25/ceramic fretting contact has been studied, in flat/flat and cross cylinders' configuration, at temperatures higher than 500°C, in order to create the glaze layer. The mechanical characterization of the tribolayer and the HS25 was performed through nanoindentation and in situ SEM microcompression experiments as a function of the temperature, in the temperature domain where the glaze layer is performant and in the temperature domain where friction and wear are important, the tests being performed in the cross section.

The first part of the presentation will be focused on the tribological contacts presentation, and micromechanical devices used description. Then, the glaze layer microstructure, chemical composition and mechanical properties will be detailed and discussed. Finally, a comparison between the mechanical properties and tribological properties of the glaze layer will be detailed [3].

This work was supported by the LABEX MANUTECH-SISE (ANR-10-LABX-0075) of University of Lyon, within the program "Investissements d'Avenir" (ANR-11-IDEX-0007) operated by the French National Research Agency (ANR).

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DEFORMATION AND FRACTURE MECHANISMS IN NANOCOMPOSITE AND NANOLAMINATE THIN FILMS REVEALED THROUGH COMBINATORIAL DESIGN AND NANOMECHANICAL TESTING

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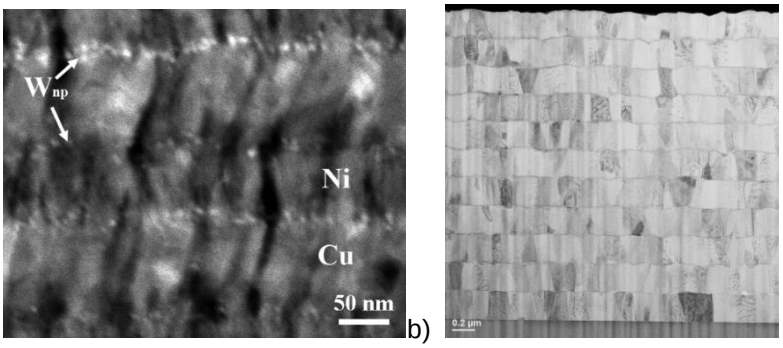
Key Words: combinatorial libraries, adhesion, nanoparticle strengthening, multilayers, thermal stability

We've integrated an atomic layer deposition (ALD), a physical vapor deposition (PVD) and a nanoparticle inert gas condensation (NP) deposition system into a single vacuum chamber. This combined system allows for PVD sputtering of micrometer thick films and incorporation of size filtered nanoparticles and/or controlled deposition of mono-layer highly conformal film coatings within a multilayer structure. In this way, unique model thin film microstructures can be architectured. We designed three thin films to understand the basic mechanism of plasticity and fracture in thin films: a) Al_2O_3 oxide films were deposited on combinatorial libraries of the ternary noble metal alloys with full compositional range to understand interfacial adhesion between oxide and noble metal alloys b) monosized tungsten nanoparticles were deposited at the interface of Cu/Ni multilayers to understand how thin film hardness and thermal stability can be engineered, c) ultrathin monolayers of Al_2O_3 layers were sandwiched between sputtered Al layers and micropillar compression was used to understand dislocation transmission and fracture across ultrathin ceramic layers.

Combinatorial libraries: By specifically programming the movement of shutters above the sample during PVD it is possible to create multilayered thickness gradients of three different materials, which can then be annealed to create films with full compositional range of a ternary phase diagram. The AlCuAu alloy consisted of multiple phases and intermetallics across the wafer; whereas the AuAgPt alloy consists of a solid-solution. Both alloys were then coated with a 500 nm thick layer of Al_2O_3, deposited using ALD, to survey the effect of composition on the adhesion probed by nanoindentation. Highest adhesion occurred in a two phase, Cu rich composition.

Nanoparticles at interfaces: The addition of nanoparticles to the interfaces in a nanolaminate, see Fig 1a, was found to increase the strength of the Cu/Ni film by more than 1 GPa above that of particle-free multilayers. However, at higher particle densities the hardness begins to decrease again, indicating there is likely an ideal particle concentration that would lead to the highest increase in hardness. This was attributed to competing mechanisms where the particles act both as dislocation sources and barriers to dislocation transmission. Initial in-situ XRD heating experiments show that the W nanoparticles can dissolve into the Ni layer to create a solid-solution Ni-W layer and react with oxygen, which could potentially create additional hardening in the material.

Ultrathin ceramic films in metal multilayers: 250 nm thick Al films have been stacked with varying Al_2O_3 interlayers (from 1 to 10 nm, in 1 nm increments, see Figure 1b.), showing high strength with a brick-and-mortar structure. 1 nm thick Al_2O_3 has been demonstrated to be sufficient for interrupting grain growth. Microcompression pillars were formed by FIB for investigation of mechanical properties. Very high yield strength was observed in the 600 MPa range. An initial TEM study of deformed samples showed that the deformation after compression is homogeneous and no obvious shearing was observed. The Al_2O_3 interlayers seems act as a dislocation sink, leading to no work-hardening and there seems no indication of dislocation accumulation at grain boundaries.



a) b)

Figure 11 – TEM cross-section of a) tungsten NP at interface of Cu/Ni multilayers and b) an Al-Al_2O_3 multilayer thin film system

MECHANICAL PHASE MAPPING OF METEORITES: COMBINING EDX AND NANOINDENTATION

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Key words: nanoindentation, meteorites, EDX

Meteorites are perhaps one of the most tangible aspects of outer space. With their irregular, ablated surfaces and intricate Widmanstätten microstructures, they truly appear to be otherworldly. The internal microstructure and composition of meteorites has been intensively studied and classified by astro- and geo-chemists to study their origins and formation processes [1]. However, other than some microhardness testing in the 1950s [2], very little has been done to characterize the mechanical behavior of these unique pieces of other worlds. With the advent of modern, high speed nanoindentation techniques, it is now possible to map the mechanical features of materials over square millimeters of area with micron-level resolution in a reasonable amount of time.

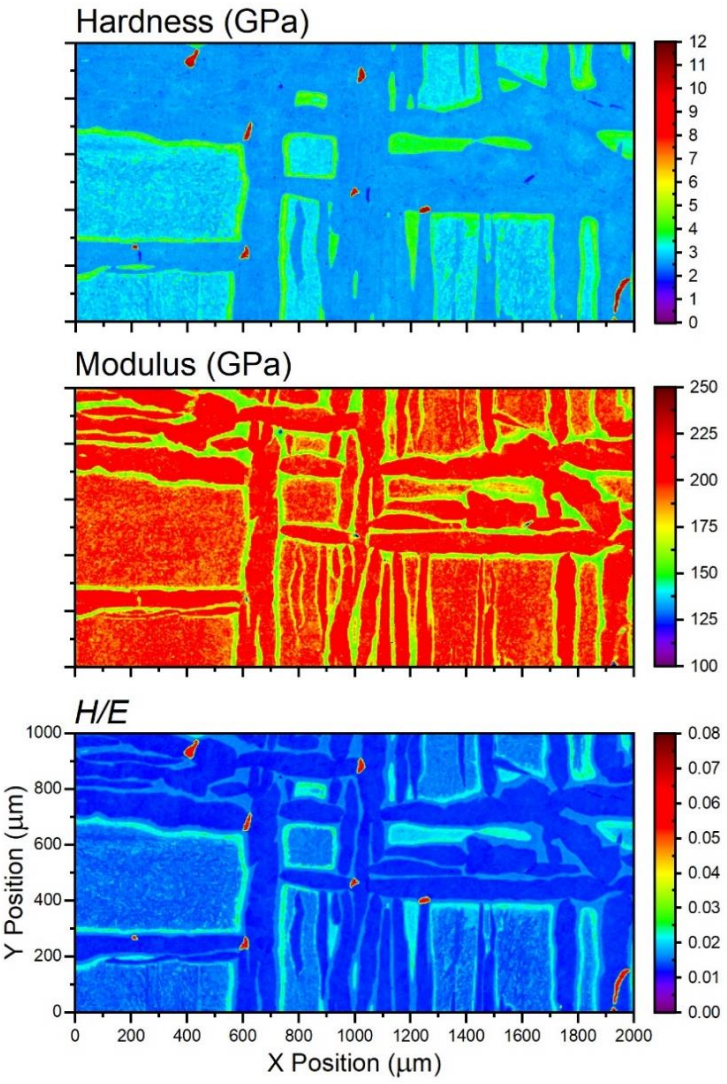

Figure 12 – Nanoindentation maps of the Hardness, Modulus and H/E ratio of a region of the Taza meteorite showing Kamacite bands in a Plessite matrix.

Nickel-iron meteorites can display a wide range of microstructures depending primarily on their nickel content, cooling rate, and tertiary composition. These range from a pure BCC iron phase (Kamacite) at low nickel concentrations to a pure FCC phase (Taenite) at high nickel concentrations. In between these concentrations, several different mixed phases occur yielding either large Widmanstätten bands of Kamacite in Taenite or a spinodally decomposed Plessite phase [1]. In **Error! Reference source not found.**, soft Kamacite bands can be see in a harder Plessite matrix in the Taza meteorite, along with several hard Schreibersite precipitates. In addition to these three phases, two additional phases can be seen to surround the Kamacite bands in the *H/E* map – TetraTaenite and the Cloudy Zone. These phases appear continuous in EDX scans, but discretely appear in mechanical phase maps.

Using statistical analysis [3], the properties of each of these phases can be extracted. By combining this information with EDX maps, the relationship between composition and properties of the various phases can be elucidated. In this work, high speed nanoindentation is combined with energy-dispersive X-ray spectroscopy (EDX) to map the mechanical properties of a variety of different nickel-iron meteorites.

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NANOINDENTATION: A POWERFUL TOOL TO EXPLORE THE WIDE CHEMICAL SPACE OF HIGH ENTROPY ALLOYS

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Key Words: nanoindentation, solid solution strengthening, high entropy alloys

High entropy alloys (HEA) are multi-component alloys, without any minor or major elements (i.e. : all elements are very concentrated) and they form a unique solid solution. It was proven that, especially for the system Co-Cr-Fe-Mn-Ni, they exist for a very wide range of composition [1]. This opens the opportunity of multi-properties optimization, like cost, density and mechanical resistance. However, to take advantage of this opportunity, accelerated mechanical testing tools are required.

In this perspective, a simplified processing route and a specific nanoindentation procedure were defined (see *Figure 13*). It permits to test experimentally 25 different HEA compositions within the Co-Cr-Fe-Mn-Ni system [2]. Tensile tests were also performed on selected compositions and were deeply compared to hardness results in order to establish a confident correlation between hardness and yield strength [3]. Based on these experimental results, it was established that (i) the solid solution strengthening (SSS) can be significantly increased by varying the composition, and that (ii) the SSS doesn't evolve linearly with composition. To go further, these experimental results were compared to a fully analytic and parameter-free model which predicts the solid solution strengthening and then the yield strength of HEA [4, 5]. The comparison between the experimental hardness and the simulated yield strength provide guidelines to appropriately choose the entry data which are needed for the model. Finally, perspectives will be drawn on HEA properties optimization.

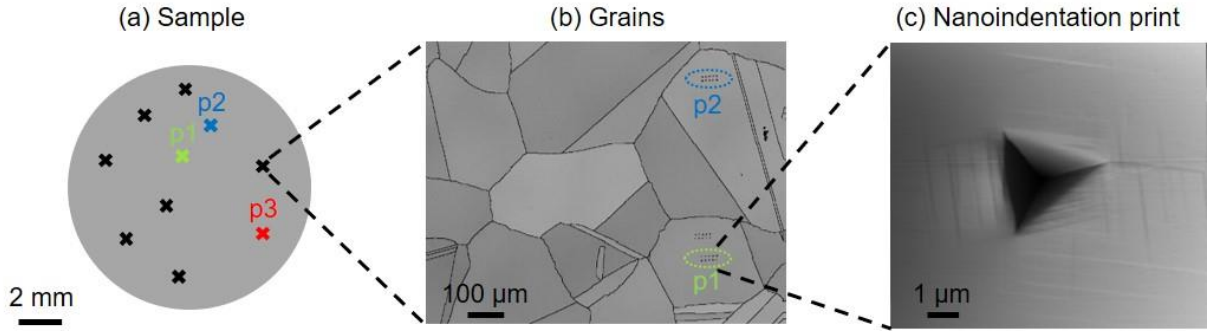

Figure 13: The nanoindentation procedure to study solid-solution strengthening of high entropy alloys.

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EFFECTS OF TEMPERTURE AND IRRADIATION DAMAGE ON FRACTURE AROUND NANOINDENTS

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Key Words: nanoindentation, fracture, FIB-SEM Tomography, Silicon Carbide

Indentation based fracture toughness measurements remain one of the fastest and most convenient ways of measuring fracture toughness and are widely used even though there are known inaccuracies with the methodologies used. In this work we use single crystal and monocrystalline silicon carbide to study the effects of temperature and irradiation damage on crack propagation and morphologies.

SiC is being widely developed as a structural material for use in the nuclear and aerospace industries in high temperature applications such as nuclear fuel cladding and combustion chamber linings for aero engines. However its lack of ductility means in must be used in the form of a SiC SiC composite. In this work we compare fracture processes in single crystal SiC and nanocrystalline SiC around Berkovich indents from RT to 750^{o}C. Hardness is seen to drop from 45GPa to 20GPa and reduced modulus from 300GPa to 200GPa across this temperature range, in good agreement with other studies. At room temperature the fracture occurs on the expected <11$\bar{2}$0> planes (fig 1). By 400^{o}C this fracture has transitioned to the <10$\bar{1}$0> planes. FIB-SEM tomography shows that there is significant changes to the subsurface cracking between the two test temperatures. Whilst at room temperature the cracks run perpendicular to the surface and link up sub surface (similar to the so called half penny cracks seen around Vickers indents), at 400^{o}C significant lateral cracking is seen with cracks running parallel to the surface. Using HR-EBSD we rationalize the change in fracture plane with a change in plastic deformation slip systems operating at high temperatures –resulting in the formation of dislocation locks leading to cracks. True fracture toughness from the known crack area are calculated to be 3MPam$^{0.5}$. This is higher than estimated form using the surface crack length alone.

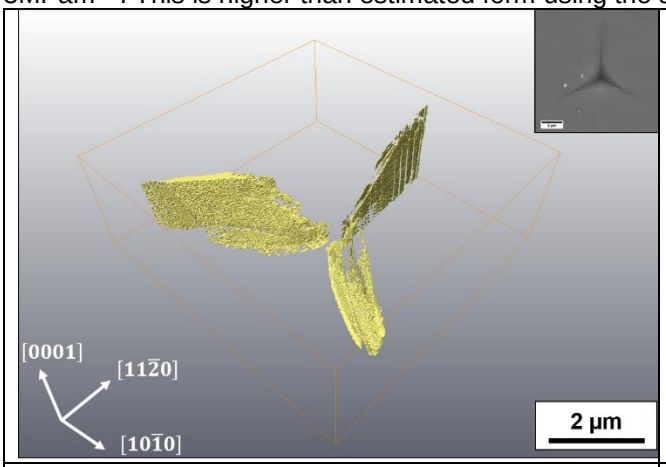

Fig 1: Cracks around nanoindents in single crystal SiC at RT. Inset shows SEM, main figures shows crack path reconstructed in from FIB-SEM tomography

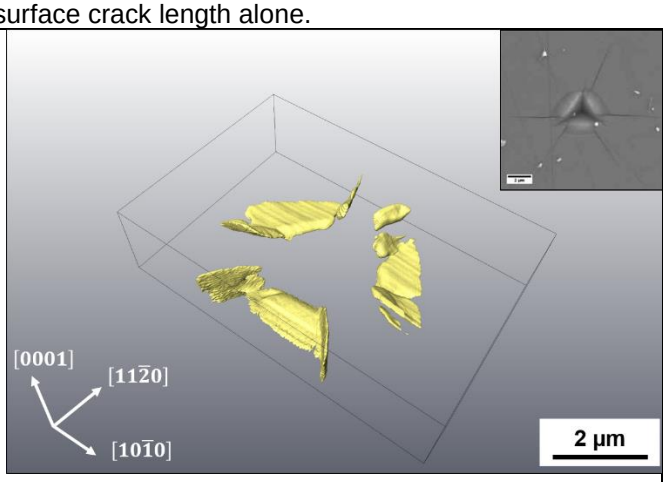

Fig 2: Cracks around nanoindents in single crystal SiC at 700^{o}C. Inset shows SEM, main figures shows crack path reconstructed in from FIB-SEM tomography

To simulate the effect of irradiation damage on the SiC samples were implanted with Ne+ and Si+ ions at 2MeV to a damage level of 2 dpa to a depth of 1.5 µm. After irradiation all cracking is suppressed in the damaged layer but large subsurface cracks are seen below this. Using HR-EBSD and Raman spectroscopy the residual stress in this layer is estimated at ~1.2GPa due to the irradiation damage. This then acts to close prevent crack formation in the damaged layer.

This results will the discussed in the context of both the advantages of using these techniques to measure fracture toughness and the limitations that are inherent to them, and new data applying this to fracture in semi-brittle graphite will be discussed.

MICROPILLAR COMPRESSION STUDY OF FE-IRRADIATED 304L STEEL

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Key Words: Austenitic steel, irradiation hardening, micropillar compression, plastic deformation, TEM.

Stainless steel used in nuclear reactors are experiencing heavy neutron irradiation that modify their microstructure, and therefore their mechanical properties. To assess the irradiation-induced hardening and the modification of deformation modes at the grain scale on 304L steels, indentations [1] and *in situ* microcompression tests were conducted on Fe-ions irradiated and non-irradiated FIB-made pillars [2]. 10 MeV and up to 8 dpa Fe irradiations were conducted at 450°C to surrogate neutron irradiation.
Size effect was detected on unirradiated but not on irradiated pillars, revealing a strong impact of the microstructure on the mechanical behavior. Surprisingly, smoother plastic deformation took place in irradiated pillars while localized shear bands were observed in unirradiated ones. TEM investigations helped elaborating some hypothesis for this different behavior.

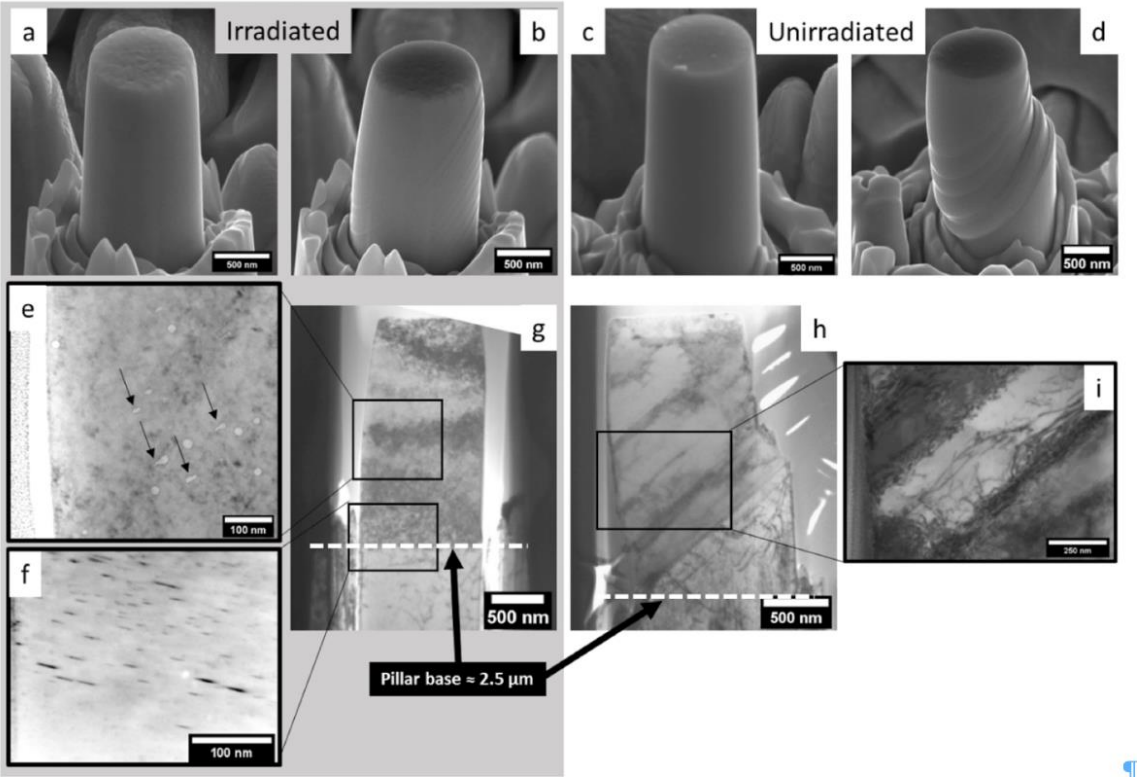

Figure 14 – Irradiated and unirradiated pillars, as FIBed, deformed to 5 and 7% respectively and observed using cross-sectional TEM

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LOCALIZED MECHANICAL PROPERTIES OF SIC-SIC FIBER COMPOSITES IN EXTREME ENVIRONMENTS – A MICROMECHANICAL STUDY

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Christian Deck, General Atomics
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Key Words: SiC-SiC fiber composites, microcantilever fracture, nanoindentation, TEM.

Silicon carbide ceramics is a promising candidate material for the use in applications where a structural material able to withstand extreme environments, in particular high temperature, is required. These include applications in aero-engines (high-pressure turbine shrouds, combustor liners and turbine nozzles, among others); in recent years there is also a considerable interest in its use in nuclear applications, in particular as an accident-tolerant fuel (ATF) cladding material. The practical use of SiC is hindered by its inherent brittleness, and therefore it is usually suggested to be used in the form of a fiber composite. Mechanical properties of the composites are largely determined by the properties of their constituents, in particular interphases but also matrix and fibers; of particular practical interest is the evolution of such properties as a function of temperature and/or radiation damage. These can be measured using micromechanical testing tools, such as nanoindentation (measuring hardness and elastic modulus) and microcantilever fracture (measuring fracture strength and toughness). In this contribution we present the results of such measurements performed in the range of temperatures, including samples that were exposed to ion irradiation. Thus, the effects of temperature and radiation are investigated and rationalized.

Material used in this study was manufactured using Tyranno-SA3 fibers in plain weave geometry, coated with pyrolytic carbon, and matrix grown by chemical vapor infiltration (CVI) method. Microstructure was investigated using transmission electron microscopy (TEM), with texture information obtained with transmission Kikuchi diffraction (TKD). It was found that both matrix and fibers are nanocrystalline, with the preferred grain growth direction in the matrix being <111>, and no texture present in the fibers. Extensive twinning was found in both matrix and fiber materials.

Temperature effects were investigated using high-temperature nanoindentation performed in vacuum at the temperatures of up to 700ºC, and cantilever testing at the temperatures of up to 300ºC. Measured values of hardness had a clear trend of decease with the increase of temperature – from 45 to 20 GPa. In the same temperature range Young's modulus decreased from 450 to 300 GPa. On the other hand, fracture toughness of fibers and matrix doesn't change significantly, but that of the interphases dramatically drops from ~0.8 to ~0.15 $MPa*m^{1/2}$. At the same time, the character of fracture changes in the interphase as well – unlike fiber and matrix at the elevated temperature, it features essentially ductile behavior.

Radiation damage was introduced via Si ion irradiation, at the temperatures of 300 and 750°C, up to the damage level of 2.6 dpa. Ion irradiation didn't lead to noticeable hardening neither of matrix, nor of fiber material. This is in contrast to the behavior of simultaneously irradiated single-crystal SiC, which showed a noticeable increase in hardness. Young's modulus at the same time decreased slightly. Fracture toughness increased in all the constituents (interphase, matrix and fiber) following irradiation, with the trend towards progressive increase with the increase of irradiation dose. More significant changes of properties in the composite compared to single crystal material was explained by the nanocrystalline nature of the composite constituents, providing high density of sinks for radiation-induced defects. On the other hand, increase of toughness is attributed to the radiation-induced near-surface stresses.

These findings are discussed in relation to their impact on the further development of SiC-SiC composites for extreme environments applications, together with the perspectives of further development of implemented methodology for such studies.

EVALUATION OF THE ENVIRONMENTAL DEGRADATION OF INTERPHASES IN CERAMIC MATRIX COMPOSITES (CMCs) VIA SEM IN-SITU MICROMECHANICAL TESTING

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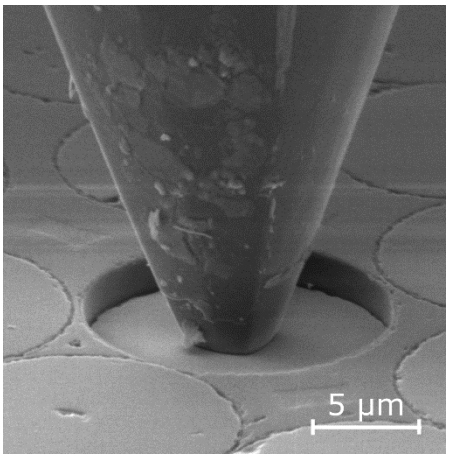
Key Words: Ceramic Matrix Composites, Interfacial shear strength, Toughening mechanisms, Interphases

The need to increase the cycle efficiency and reduce NOx emissions from aero-engines has promoted the development of Silicon Carbide (SiC) based Ceramic Matrix Composites (CMCs) which have entered in service in aircraft turbine engines as replacements for some Ni-based superalloys. The main tendency of material choice is converging to CMCs constituted by SiC fibres coated with a thin (0.1-1 µm) BN interphase within a SiC matrix (SiC/BN/SiC), resulting in an optimised tough ceramic composite. However, unlike the generic tendencies found for metallic materials, environmental effects seem to not follow a clear tendency as hottest temperatures do not necessarily result in more severe degradation. This is due to the complex degradation thermodynamics occurring at the interface of the SiC-BN system such as volatilisation of B species, borosilicate glass formation or formation of self-healing oxide products.



The present study focuses on understanding how the interfacial and fibre properties in SiC/BN/SiC are affected by different aero-engine inspired degradation cycles by exploring the interfacial properties via push-out tests and the fibre properties via three point bending of single fibres. These tests are performed in-situ to achieve a higher repeatability in the testing conditions and allowing for characterization of the variability coming from the ceramic material itself. These mechanistic results are then complemented with TEM-based characterization techniques in order to link the change in properties to the changes in chemistry and microstructure.

The goal of this work is then to understand how the localised changes appearing mainly at these interphases affects the overall toughening behavior of SiC-based CMCs at the bulk scale.

Figure 15 – SEM in-situ push out test on a SiC/BN/SiC CMC

ELEVATED TEMPERATURE NANOINDENTATION AND IN-SITU SEM MECHANCIAL TESTING OF URANIUM FUELS

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Key Words: High Temperature Nanoindentation, Advanced Nuclear Fuel, Ceramics, In-situ SEM testing, Microcompression Testing

Due to the Fukushima nuclear accident there has been a large effort by several countries to develop accident tolerant fuel forms for commercial light water reactors. A challenge with the current UO_2 fuel is its low thermal conductivity which leads to higher center line temperatures in the fuel. New nuclear fuel forms are looking to increase the thermal conductivity and other thermophysical proprieties while also maintaining adequate mechanical properties and uranium loading. The elastic modulus, fracture toughness, and creep properties of the fuel are important for modeling the pellet clad mechanical interactions during operation of a nuclear reactor. During the operation of a nuclear reactor the cladding material creeps down and fuel pellet swells which leads to physical contact between the two. The pellet clad mechanical interactions can lead to potential cladding failures and release of radioactive material. The advanced fuel forms that are under consideration for replacing UO_2 in commercial light water reactors is UN, U_3Si_2, composite UO_2 and UO_2 with additives. The composite UO_2 is looking to increase the thermal conductivity with different additions and the UO_2 with additives are intended to increase the grain size of the UO_2. The increase in grain size can reduce the release of fission gas products into the plenum of the cladding rod improving the operational lifetime of the fuel. While there is a large amount of work on the thermal properties of these accident tolerant fuel forms the literature is quite sparse on the mechanical properties necessary for modeling such interactions as the pellet clad mechanical interactions.

A technique that could measure the mechanical proprieties like hardness, elastic modulus, fracture toughness and creep properties of these new materials in their operating temperature range is elevated temperature nanoindentation. The periphery of fuel pellets in a light water reactor operates at 500 °C which is within the temperature range of current commercial high temperature nanoindentation systems. The main challenge with elevated temperature nanoindentation of these uranium base compounds is their sensitivity to oxygen. These uranium based compounds readily oxidize at the elevated temperatures (>500 °C) without environmental control in the nanoindenter chamber. For this study, a Hysitron Triboindenter has been modified to perform high temperature nanoindentation in an inert or reducing environment, minimizing oxidation in the specimens and facilitating the measurement of mechanical properties. The data collected will provide valuable datasets that feed directly into models for understanding the behavior of these advanced accident tolerant fuel forms in light water reactors.

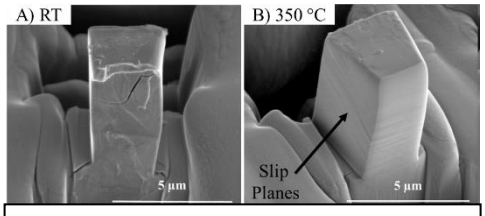

Figure 1: A) An in-situ SEM microcompression test of UO_2 at room temperature B) A microcompression test at 350 °C

In addition, in-situ scanning electron microscopy testing at the micron scale is being investigated as an addition way of measuring the mechanical properties of UO_2 and these advanced accident tolerant fuel forms. The development of these small scale mechanical testing techniques on fresh fuel would allow for applying them to spent nuclear fuel in the future. This would be of great interest in nuclear community since there is limited mechanical data of spent fuel in the literature due to the difficulty of testing the material because of its high levels of radioactivity. Microcompression testing in a single crystal of UO_2 at room and elevated temperature has been performed using the low vacuum mode of FEI Quanta 3D FEG SEM/FIB dual beam system at UC Berkeley. A Hysitron PI-88 system was used to perform the microcompression testing of the FIB manufactured specimens. During the testing a brittle to ductile transition in the deformation behavior was observed (as seen in Figure 1) and the Peierls stress for UO_2 was calculated that agreed well with the literature data. In addition, the microcompression specimens allow for the calculating the slip systems activated in the UO_2 at these temperatures. The successful results on the UO_2 allows progressing with in-situ SEM testing of the advanced accident tolerant fuel forms at elevated temperatures.

MEASURING NANOINDENTATION HARDNESS AT HIGH SUSTAINED STRAIN-RATES

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Key Words: Nanoindentation, Strain-rate, Continuous stiffness measurements

Dynamic pyramidal nanoindentation, performed at a constant strain-rate, is a popular nanomechanical method for accessing the local strength of complex materials. However, with currently available testing systems using continuous stiffness measurement (CSM), nanoindentation is so far limited to strain-rates of $\sim 10^{-1}$ s^{-1}, which precludes it from ballistic applications. Here, we show that the current limitation derives primarily from a plasticity issue related to the continuous stiffness measurements.

Increasing the harmonic frequency would allow for a corresponding extension of the strain-rate range. Unfortunately, this is usually prevented by machine resonance effects, which become prominent above several multiples of the natural frequency of the measurement system. Here, we show the existence of so-called “sweet spots” frequencies, one order of magnitude higher than the standard harmonic oscillation, which are nonetheless unaffected by undesirable resonance phenomena and can be selected for performing valid hardness measurements.

In order to access even higher deformation rates, we also show how the Oliver-Pharr evaluation method can be modified, so as to avoid the need for a measurement of the contact stiffness. Specifically, provided the elastic modulus is known from a prior measurement, the hardness can be calculated as a function of the indentation depth from quasi-static indentation loading data. As the new evaluation method strongly relies on the load and displacement input data, the experimental upper strain-rate limit is mostly determined by the time constants of the hardware components in the nanoindentation system. With most current commercial systems, valid measurements are shown to be possible up to strain-rates of $\sim 10^{+1}$ s^{-1}.

These improvements enable the characterization of the small-scale mechanical properties of materials over a much larger strain-rate range than previously achievable with standard CSM testing, as will be illustrated by an application to the superplastic alloy Zn22Al.

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IMPACT OF TEMPERATURE AND HYDROGEN ON THE NANOMECHANICAL PROPERTIES OF A HIGHLY DEFORMED HIGH ENTROPY ALLOY

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Due to their quite attractive properties, high-entropy alloys have emerged to an intensely studied class of alloys within the past years. Besides their high strength and maintained ductility, literature reports modest sensitivity to hydrogen embrittlement for conventional microstructures. Utilizing severe plastic deformation methods, for example high-pressure torsion, it is possible to further tailor the mechanical properties by microstructure refinement to the nanometer regime, which in turn increases the hydrogen storage capability at internal defects and boundaries. Furthermore, the nanocrystalline grain size provides markedly enhanced strength values, while the high fraction of grain boundaries influences the hydrogen diffusion and storage kinetics.

Within this study, the micromechanical characteristics of pure Ni and a single phase face-centered cubic CrMnFeCoNi alloy in fine and ultra-fine grained microstructural conditions, fabricated by high pressure torsion, will be investigated in detail. Moreover, electrochemical in-situ nanoindentation will be employed to determine the impact of hydrogen charging on the mechanical performance of this high-entropy alloy class and will be set into context to result found for pure Ni.

STUDYING DEFORMATION MECHANISMS OF NANOCRYSTALLINE NICKEL BY THERMAL ACTIVATION ANALYSIS AT SUBAMBIENT TEMPERATURES AND HIGH STRAIN RATES

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Key Words: Thermal activation, nanocrystalline Ni, micropillar, high strain rate, cryogenic temperature

Electrodeposition and magnetron sputtering are promising methods for depositing thin films with nanocrystalline (nc) microstructures. Nc metals are attractive materials, as they show considerably higher mechanical strength compared to their poly- or monocrystalline counterparts. However, they also feature pronounced time- and rate-dependent inelastic behavior and their microstructure may change drastically when exposed to elevated temperatures or ion irradiation. Therefore, in order to assess the mechanical behavior and deformation mechanisms of these materials under controlled conditions and at a constant microstructure, it is desirable to perform thermal activation analysis at subambient temperatures and high strain rates on pristine samples.
Large arrays of micropillars were fabricated by electrodeposition of nc Ni into lithography molds by LIGA leading to non-tapered, damage-free microspecimens. X-ray diffraction (XRD) measurements and transmission electron microscopy (TEM) imaging revealed a grain size of approximately 28nm. EDX analysis showed a homogeneous elemental composition and no concentration of impurities at the grain boundaries. A micromechanical testing device was developed that allows performing nanomechanical experiments at sub-ambient temperatures down to 120K in a large range of strain rates between 10^{-4} and $10^{3}s^{-1}$.
Micropillar compression experiments were performed on the nc Ni micropillars in a scanning electron microscope (SEM) at temperatures ranging between 160K and 293K and strain rates from 10^{-3} to $5{\cdot}10^{2}s^{-1}$. Yield stress was extracted based on a 2% strain offset rule and found to vary strongly with temperature. Post-experimental high resolution images were taken in a SEM to reveal deformation patterns. Furthermore, TEM lamellae were prepared from micropillars tested at strain rates of $10^{-3}s^{-1}$, $25s^{-1}$, and $500s^{-1}$ and imaged to reveal deformation-induced changes in microstructure. Strain rate sensitivity exponent m was determined to be in the range of 0.003 to 0.008. Apparent activation volume was found to decrease with temperature from $10b^3$ at 293K to $2\text{-}4b^3$ at 160K. These values are consistent with dislocation nucleation, but also other deformation mechanisms like grain boundary or defect diffusion.
This study highlights a new approach for assessing the mechanical behaviour of nc materials by testing large numbers of pristine electrodeposited micropillars at various subambient temperatures and in a large strain rate range. This allows assessing the deformation mechanisms by thermal activation analysis while keeping artifacts from specimen preparation and microstructural changes throughout the experiments at a minimum.

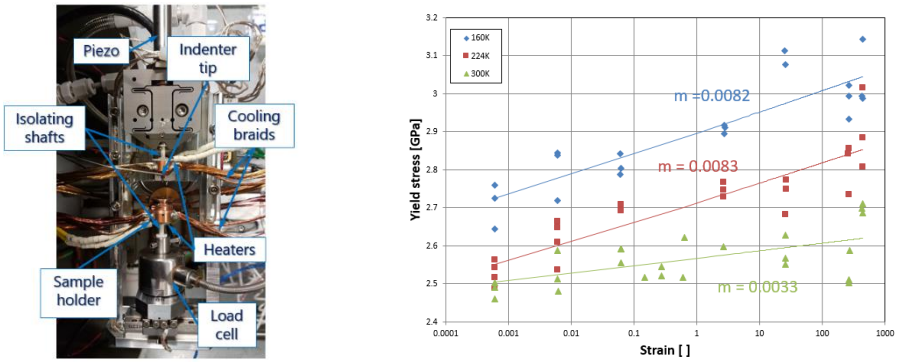

Figure 16 – Left: Cryogenic indenter setup. Right: Yield stress as a function of temperature and strain rate

HYDROGEN-MICROSTRUCTURE INTERACTIONS BY NOVEL BACK-SIDE HYDROGEN CHARGING DURING *IN SITU* NANOINDENTATION

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Key Words: Hydrogen, *in situ* nanoindentation, Fe-based alloys, micromechanics.

To understand how hydrogen interacts with different features (e.g. dislocations, grain boundaries, precipitates, etc.) in alloys and composites is essential either to control and benefit from the hydrogen technology, or to prevent the destructive outcome of hydrogen embrittlement. Failure mechanisms initiate at the atomic scale with hydrogen absorption and further interaction with trap binding sites or defects. Nanoindentation and related techniques are valuable tools to study independently such mechanisms due to the small volume probed. Even more, *in situ* testing while charging the sample with hydrogen can prevent the formation of concentration gradients due to hydrogen desorption.

Two custom electrochemical cells were built for *in situ* hydrogen charging during nanoindentation of the sample (Figure 1): “front-side” charging with the sample and indenter tip immersed into the electrolyte, and “back-side” charging where the analyzed region is never in contact with the solution and therefore the observed effects are only due to hydrogen. We discuss the advantages and disadvantages of both approaches during the study of the hydrogen effect on the mechanical behavior and incipient plasticity in bcc FeCr alloys. A reduction in the pop-in load indicating the yield point with the increase of hydrogen content and formation of multiple pop-ins during nanoindentation provided evidence for the decrease in the resolved shear stress and enhanced dislocations nucleation. This behavior is consistent with multiscale simulations of homogeneous dislocation nucleation where the reduced critical shear stress can be explained as an effective decrease of the dislocation line energy due to the interaction with diffusible hydrogen.

The newly developed back-side charging technique, in addition, allows overcoming surface degradation that might occur during front-side charging and possibly reducing the reliability of post deformation analysis. The presence of hydrogen on the top analyzed surface (Figure 1b) was assessed by Kelvin probe measurements, showing a fast hydrogen diffusion rate towards the upper surface as well as a pronounced release flow for the analyzed Fe-Cr alloys. Furthermore, during back-side charging, a hardness increase was observed while rising the hydrogen activity at the entry side, consequently increasing the hydrogen content. This is attributed to an enhanced dislocation nucleation that might concurrently suppress dislocation mobility.

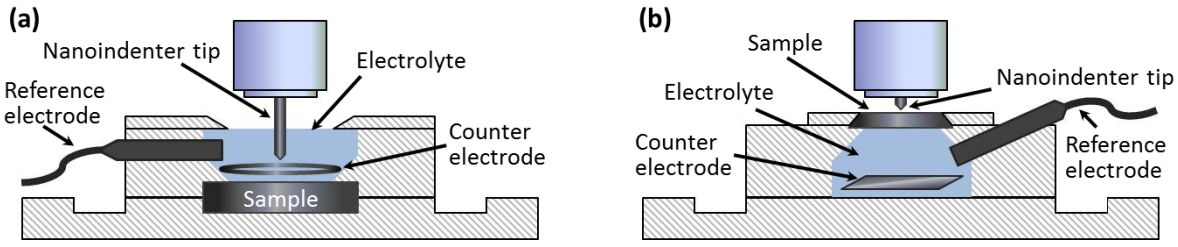

Figure 1 – Schematic of the built setups for in situ nanoindentation during (a) front-side hydrogen charging, and (b) back-side hydrogen charging.

A NEW NANOINDENTATION CREEP METHOD USING CONSTANT CONTACT PRESSURE

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Key Words: Strain rate sensitivity, nanocrystalline alloys, dislocation mechanism

A new indentation creep testing method is proposed where the mean contact pressure is kept constant. An approach of the proposed method is quite different from conventional indentation creep experiments, where the load on the sample is kept constant and the hardness is constantly decreasing due to the increasing contact depth / area. In this newly developed constant contact pressure method (CCP), the mean contact pressure as defined through Sneddon's hardness is kept constant, until a steady state strain rate is achieved. Besides controlling the mean contact pressure, the dynamic stiffness is furthermore used to assess the indentation depth, minimizing thereby thermal drift influence and pile-up or sink-in effects during long-term experiments. The CCP method has been tested on strain rate sensitive ultrafine grained (UFG) CuZn30, UFG CuZn5 as well as on fused silica, comparing the results to strain rate jump (SRJ) tests as well as to the CLH nanoindentation creep tests. With the CCP method strain rates from $5x10^{-4}$ s^{-1} down to $5x10^{-6}$ s^{-1} can be achieved, keeping the mean contact pressure constant over a long period of time, in contrast to the CLH method. Moreover at low contact pressures, the strain rate sensitivity exponent of the tested materials is strongly increasing. There the plastic zone is only slightly increasing an internal relaxation processes within the plastic zone dominate the deformation behaviour. The CCP technique thus offers new possibility of performing long-term creep experiments while retaining the contact stress underneath the tip constant.

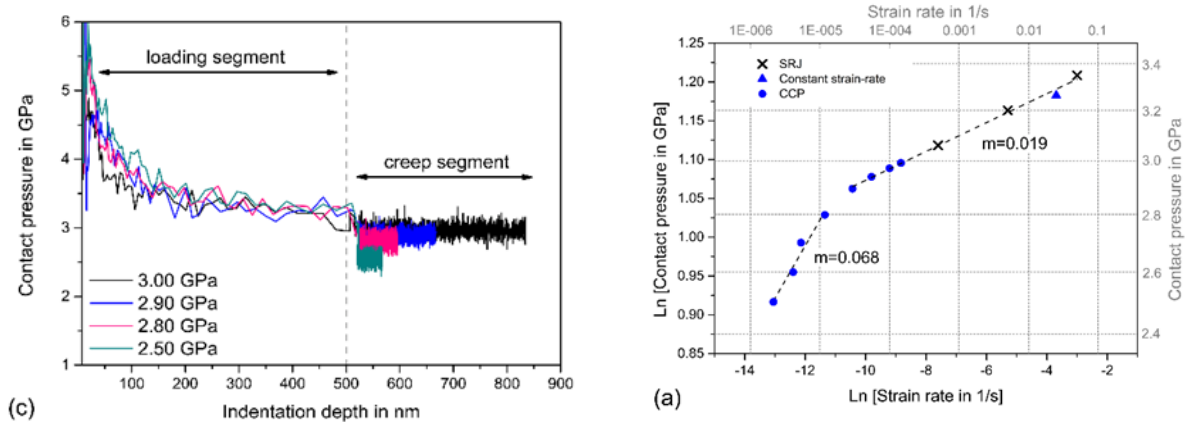

Figure 17 – left: Contact pressure vs. indentation depth, right: contact pressure vs. indentation strain rate

INDENTATION CREEP TESTING OF SUPERALLOYS

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Key Words: Indentation creep testing; solid solution strengthening; high temperature indentation, creep testing; superalloys

Great progress has been made over the last years in high temperature nanoindentation testing and quite reliable test systems are available to operate at temperatures up to 800°C. With such systems the high temperature strength is measured via the hardness of materials. However, for high temperature materials especially the creep strength is of interest and therefore also many attempts have been undergone to probe also the creep properties with high temperature nanoindentation. In most cases pointed indenters as Berkovich or conical indenters have been used for this. A major challenge, however, then is, how the nanoindentation data are converted into uniaxial creep properties, i.e. those which are needed for constructional purposes. Although, it seems that the stress exponent can be derived quite successfully with such indenters, an evaluation of a full creep curve for materials with significant primary creep does not seem possible, since the strain a pointed indenter is inducing is fixed by the indenter shape and stays more or less constant during the whole test [1].

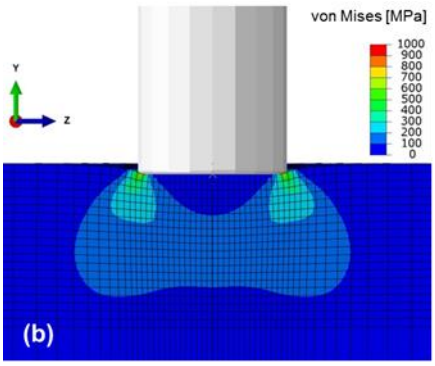

Fig. 1: Stress field underneath a flat punch indenter, from [3].

Therefore, flat-punch indenters are quite attractive, where this problem does not occur. In the literature many examples can be found, where indentation creep testing with a flat-punch indenter has been used successfully, going back to the early work of Yu and Li from 1977. However, in most cases flat-punch indenters with quite large diameters have been used, which do not allow local measurements. Recently, a new indentation creep testing approach has been developed which uses a flat punch indenter with a diameter of only 20 µm [3]. In the new set-up the creep experiments are performed inside a thermo-mechanical analyzer (TMA) which provides an excellent temperature stabilized environment for temperatures up to 1200°C and any possible drift effects can be completely neglected.

The method has been validated on single crystalline Ni alloyed with Re, Ta or W at a temperature of 650°C. Using crystal plasticity finite element modeling, the indentation creep response is converted into equivalent uniaxial creep properties. It is shown that the conversion parameters, evaluated for differently oriented single crystals, can be chosen independently of the creep rate exponent in the power law creep regime. It is found that the indentation creep results agree well with conventional uniaxial creep tests. Furthermore, the indentation creep testing setup has been used to study locally the creep rate of dendritic microstructures in Ni-base superalloys at 750°C. It is found that the creep strength in the dendrite core is significantly higher than in interdendritic regions, which corresponds with the enrichment of the most important strengthening element Re there.

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MEASUREMENT OF THE CREEP BEHAVIOR OF THIN ZRNI METALLIC GLASS FILMS – A COMPARISON BETWEEN NANOINDENTATION RELAXATION, NANOINDENTATION CREEP AND LAB-ON-CHIPS EXPERIMENTS

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Key Words: Time-dependent behavior, Nanoindentation relaxation, Amorphous materials, Finite element modelling.

The characterization of the time-dependent behavior of thin metallic glass films is one of the key-issue for surface engineering. Such a measurement requires loading a constant material volume located in the thin film. Unfortunately, this condition is not fulfilled in the commonly used creep nanoindentation testing, contrary to micro tensile lab-on-chip experiments or micropillar compression testing. In this paper, we show that nanoindentation relaxation is an efficient alternative to nanoindentation creep. For that purpose, an extensive study of ZrNi metallic glasses viscoplastic behavior is performed using several experimental set-up (lab on chips, nanoindentation relaxation, nanoindentation creep, constant strain rate, ...). An innovative nanoindentation methodology is used to perform long-term relaxation tests up to 10 h with excellent reproducibility. It consists in maintaining a constant contact area during the test by controlling the contact stiffness between the tip and the material. Nanoindentation relaxation, constant strain rate loading and lab-on-chips data lead to similar values of apparent activation volume and strain rate sensitivity, whereas nanoindentation creep clearly overestimates the activation volume (Fig 1). Finite element modelling of nanoindentation creep and nanoindentation relaxation also confirms this trend. We evidence, thanks to the long-term indentation relaxation test that the underlying deformation mechanisms remain unchanged on the entire investigated strain rate range.

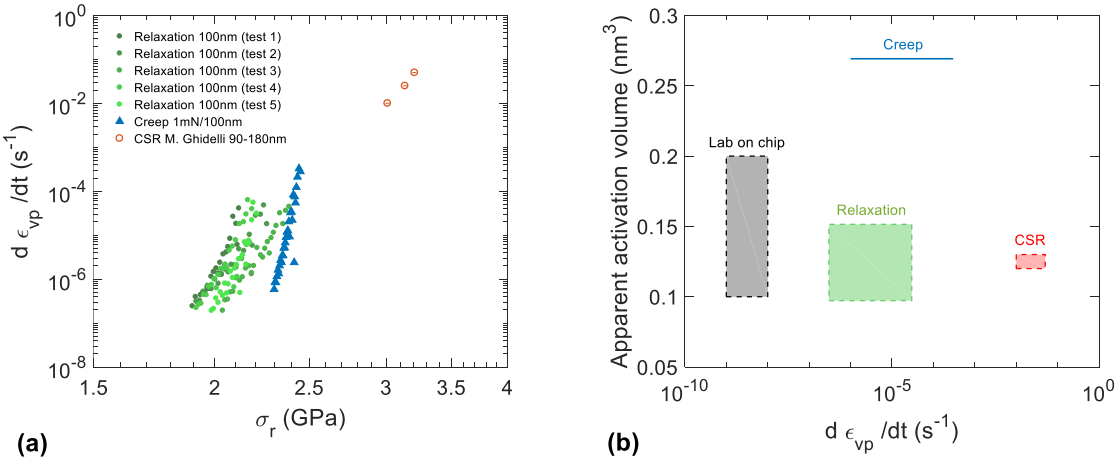

Figure 18 – Viscoplastic behavior of 900~nm layer ZrNi metallic glasses. (a) strain rate versus representative stress as measured by nanoindentation relaxation, creep and constant strain rate experiments. (b) Apparent activation volume from nanoindentation and lab-on-chip experiments as a function of viscoplastic strain rate.

DIRECT OBSERVATION OF YIELD IN FILMS BY FLAT PUNCH INDENTATION

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Key Words: flat punch, uniaxial compression, yield point, constitutive properties, polymer film

In regular indentation many strain states are simultaneously present in the indented region, so measured parameters such as hardness and modulus are average values over a wide range of strains. Testing of structures such as pillars, levers or film bulges enables determination of yield point and modulus with uniform strains in the sample, but requires specialised sample preparation and can be significantly affected by surface condition.

Here we show how in-situ indentation with a flat punch allows direct observation of a discrete yield point in soft films on more rigid substrates. The yield point is clearly observable from the load displacement behaviour and from post indent AFM imaging. The film is in uniform uniaxial strain. Finite element simulations show that effective self-confinement by surrounding film material leads to uniformity throughout the film material down to surprisingly low aspect ratios around 4:1. This occurs for a significant range of stresses above the yield point. Eventually at even higher stresses the film material is extruded laterally.

The characteristics of the yield event will be described as a function of temperature and film thickness for thin to ultrathin films. At higher aspect ratio and with sufficient stiffness of punch and substrate, quantitative, in-situ measurement of intrinsic stress vs. strain to well beyond the elastic limit becomes possible for thin films. The extent to which full constitutive relations for polymer films can be determied will be discussed, along with limitations of the technique.

MEASUREMENT OF HARDNESS AND ELASTIC MODULUS BY DEPTH SENSING INDENTATION: FURTHER ADVANCES IN UNDERSTANDING AND REFINEMENTS IN METHODOLOGY

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Keywords: nanoindentation, hardness, elastic modulus, stiffness

Depth sensing indentation technique has been widely used to measure small scale mechanical properties over the years. Starting from the seminal work of Oliver & Pharr [1], there have been many improvements / modifications to the test methodology and also significant advances in measurement electronics / testing instrumentation. These advancements provide opportunities to not only develop novel testing capabilities but also further improve the precision and accuracy of the most common measurement parameters – hardness and elastic modulus.

In this regard, this work presents a comprehensive study on the various steps involved in a typical depth sensing indentation test, viz., surface approach, surface detection, load-time history including superimposing an oscillatory force on broad band load, unloading and drift rate measurement. The effect of each of these steps on the accuracy and precision of the hardness and elastic modulus measurement will be discussed with specific focus on frequency specific testing techniques such as continuous stiffness measurement. The effect of the instrument's measurement time constants and dynamic parameters such as mass, spring constant and damping coefficient during different steps of an indentation test and thereby on the hardness and elastic modulus will be presented. A simple model is developed to simulate a depth sensing indentation test that incorporates the material and instrumentation parameters to help visualize the overall process and provide new insights for pushing the limits of the currently available instrumentation for improved precision and accuracy. This involves performing tests beyond the traditional boundaries of parameter space such as increased oscillation amplitude, strain rate, oscillation frequency, etc. For instance, if the indentation strain rate gets high compared to the oscillation frequency, inaccuracies can occur. This work presents the critical experimental parameters and the associated first order corrections for the potential errors. The model predictions and corrections are validated on different classes of materials. Finally, guidelines for measuring hardness and elastic modulus using a depth sensing indentation test with significantly improved precision and accuracy within the limitations of the currently available instrumentation will be discussed.

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A NEW APPROACH TO EVALUATE RESIDUAL STRESS USING INSTRUMENTED INDENTATION TESTING AT NANO SCALE

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Keywords: Residual stress, Indentation, Load-displacement curves

In structural integrity, residual stress is one of the major factors affecting structure failure. In particular, tensile residual stress accelerates crack growth and reduces integrity. Hence test methods have been devised that can quantitatively evaluate residual stresses, including X-ray diffraction, hole-drilling, and contour methods. Now a relatively new technique, instrumented indentation testing, can be used to quantitatively evaluate the surface residual stress of a structure semi-nondestructively with mechanical response causing small indents. Many studies have confirmed that indentation load-displacement curves are shifted depending on the residual stress state. For the same indentation depth, a larger indentation load is required for a compressive residual stress state, and a smaller indentation load is required for a tensile residual stress state, in contrast to the stress-free state. Thus, for the same indentation depth, there is a difference in indentation load between the stressed and stress-free states. Kwon and Lee have suggested and verified experimentally that, among the surface residual stress components, a deviatoric stress term parallel to the indentation axis induces a virtual force that affects the plastic deformation occurring during indentation, and consequently also affects the indentation load-displacement curve. [1] In this paper, principle and application for measuring residual stress by IIT at multi-scale will be included.

References

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PASSIVE AND ACTIVE MECHANICS OF BANKSIA SEED PODS

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Key Words: biological and bioinspired materials, hierarchical structure, multifunctional, experimental micromechanics, seed pods

Biological materials consist of only a few basic building blocks, namely sugars, proteins and a few minerals which are assembled into structurally complex materials to ensure (multi)functionality for the particular organism. Prominent examples are bone or nacre, composites of mineral and protein possessing high stiffness and strength. Apart from mineralized materials, protein- and sugar-based materials such as spider dragline silk or plant fibres achieve similarly high values. On the other end of the scale are soft materials with 3 to 4 magnitude lower stiffness and strength values (eg. parenchymatic tissue or artery). Common concept for all biological materials is that a wide range of material properties is achieved by structuring rather than changing their chemical components and frequently materials combine high strength and toughness when needed.

A comprehensive understanding of the structure function relationships of biological materials requires measurements of mechanical properties at a range of different length scales, often in combination with other techniques (eg. X-rays, microscopy, spectroscopy). This approach will be illustrated on the example of plants and especially dead but multifunctional tissues such as the seed pods of *Banksia attenuata*, a native Australian species. The seed storing pods can remain on the plant for up to 15 years without metabolism before they open upon elevated temperatures (eg. caused by bush fires). During the storage period the seed pod material must passively resist weathering, microbial degradation and attacks by bird beaks. Interestingly, the seed pods do not open at uniform temperatures. Instead, opening temperatures change gradually along a climatic South-North gradient increasing towards North. We were able to identify the “temperature sensor” of the seed pods: the inner curvature of the layered follicles gradually increases providing Northern seed pods with a higher flexural rigidity. Opening is activated by a temperature-dependent decrease of the elastic modulus of the inner resistance layer, allowing pre-stresses to be released. However, the initial opening is not sufficient to release the seeds, further opening is fueled by moisture changes which lead to directional swelling and at the same time to changing mechanical properties in different layers of the seed pod.

The findings on Banksia seed pods provide inspiration for self-sensing, moving and actuating materials and systems. We expect a comparably easy transfer into technical application because metabolism and biological signaling is not required for functionality. Since the material consists only of a few basic building blocks, namely cellulose, hemicelluloses, lignin, tannins and waxes, recycling and sustainable material use seem to be much easier compared to multi-component composites.

SMALL SCALE FRACTURE OF BONE TO UNDERSTAND THE EFFECT OF FIBRILLAR ORGANIZATION ON TOUGHNESS

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Key Words: Bone, Fracture, SEM, In situ

Fracture toughness is a critical component of bone quality and derives from the hierarchical arrangement of collagen and mineral from the molecular level to the whole bone level. Molecular defects, disease, and age affect bone toughness, yet there is currently no treatment to address deficits in toughness. Toughening mechanisms occur at every length scale, making it difficult to isolate the influence of specific components. Most experimental studies on the fracture behaviour of bone use milled samples of bone or whole bones. Toughness deficits can be identified but may be caused by a multitude of parameters across length-scales, making it difficult to develop targeted therapies. Herein, we measure the toughness of bone in micropillars where porosity and heterogeneities are minimized, allowing us to determine the role of fibril anisotropy on fracture toughness. Double cantilever beam micromechanical tests were conducted in a scanning electron microscope on 4x6x15 mm pillars of mouse bone femorae produced in the longitudinal and transverse orientations. Subsequent transmission electron microscopy of the fractured pillars revealed a role of the local organization of the mineralized collagen fibrils in influencing crack propagation. We demonstrate that fibril orientation is a critical factor in deflection during crack propagation, significantly contributing to fracture toughness.

MICROTENSILE PROPERTIES AND FAILURE MECHANISMS OF BONE ON THE LAMELLAR LEVEL

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CORRELATION OF ULTRA-FINE REAL-GEOMETRY FEM MODELS OF DIATOMS DERIVED FROM NANO-XRAY TOMOGRAPHY WITH IN-SITU NANOMECHANICAL TESTING

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Key Words: In-situ nanoindentation, diatoms, frustules, Finite Element Analysis, nano-X-ray tomography

Diatoms are unicellular, photosynthetic microalgae with complex hierarchical shell morphologies and features. The unique, three-dimensional anatomy of their silica exoskeletons (frustules) contain structure features ranging from the nano-, submicro- to the micrometer-scales (Figure 1). Due to their extraordinary properties, these frustules have drawn attention from a variety of research fields and they have been proposed to be used in a range of applications, including templates for drug delivery carriers, oil and water separation membranes, optical devices, metal alloy components as well as metamaterials designs. Several studies have shown that diatom frustules show unique mechanical properties such as high specific strength and resilience against fracture. Most of these properties arise from the hierarchically arranged structural features.

In this work, we will show in-situ nanoindentation experiments on specific positions of single diatom frustules. Nano-X-ray computed tomography (nXCT) imaging has been conducted at exactly the same frustule structures. Based on this 3D nXCT data ultra-fine real geometry Finite Element twins of the frustules have been created. Consequently, the real in-situ nano-indentation experiments have been repeated at the digital FEM twin of the experiment. The analysis and comparison between experiment and simulation then was used to shed light on the mechanics of didymosphenia geminata frustules.

The method described in this study holds great potential to explain how morphology is pivotal to the mechanical performance of the frustules' hierarchically arranged structures and provides helpful insight for the design of damage-tolerant lightweight engineering materials.

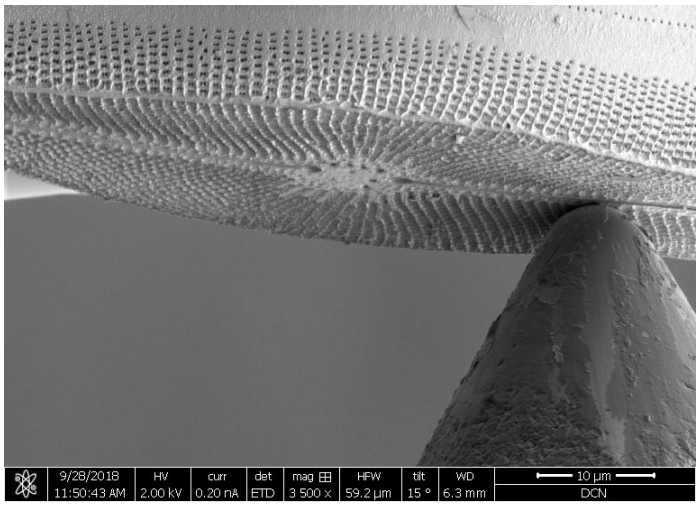

Figure 19 – In-situ nanoindentation experiment at a diatom frustule (SEM)

HIGH STRAIN RATE PLASTICITY IN MICROSCALE GLASS

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Key Words: High strain rate, micropillar, amorphous, shear transformation zone, plasticity

Understanding the materials behavior at high strain rates is critical for the design of structures subjected to accidental overloads such as crash testing of vehicles and impact resistance of surface coatings. From a scientific perspective, experimental determination of high strain rate properties at the micro- and nano-scale will allow the bridging of time scales between atomistic simulations and experiments, leading to a direct comparison between the two methods. Despite many efforts to expand the range of micro and nanomechanical testing in terms of forces, temperatures and loading conditions, the achievable strain rates are still around $10^{-5}\,s^{-1}$ to $10^{-2}\,s^{-1}$. This limited range of strain rates is primarily due to lack of testing platforms capable of simultaneous high-speed actuation and high-speed sensing of microscale displacements and millinewton loads. This presentation will report, a piezo-based experimental methodology for conducting high strain rate *in situ* micropillar compression testing at rates upto ~2000/s inside a scanning electron microscope (SEM), including a brief overview of the advantages and challenges of microscale high strain rate testing compared to traditional macroscale, Kolsky bar based, high strain rate testing.

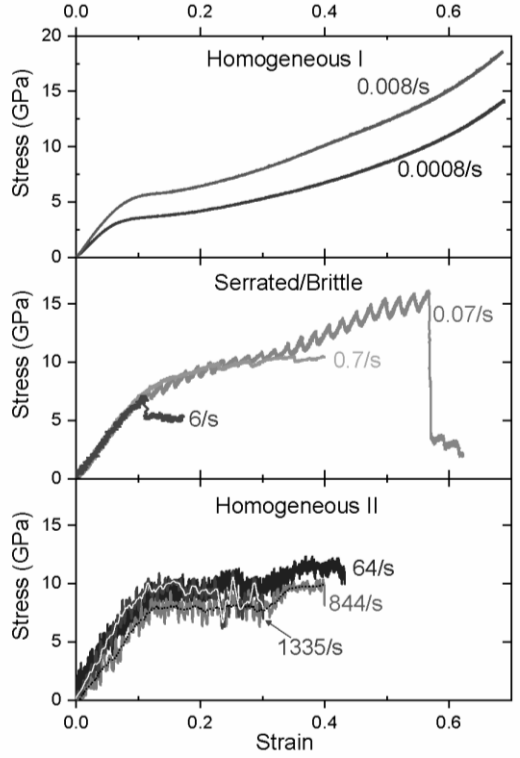

Figure 1: Rate-dependent stress-strain response of microscale glass

Glass is a ubiquitous material with recent applications in the MEMS industry as a more robust alternative to silicon in applications such as high frequency resonators and actuators. Such applications of microscale glass require a clear understanding of their dynamic properties to assess their reliability and crashworthiness. But the mechanical properties of microscale glass are known only at quasi-static strain rates (<~0.1/s). In this presentation, the deformation and failure of microscale glass will be presented as a function of strain rate across 8 orders of magnitude upto 1330/s, using *in situ* micropillar compression tests in an SEM. Contrary to macroscopic experiments, glass micropillars show a remarkable ductile-serrated-brittle-ductile transition in deformation behavior as the strain rate is increased from 0.0001/s to 1335/s, as shown in Figure 1. Further, the atomistic mechanisms behind such dynamic plasticity behaviors of glass will be explained using a combination of high-resolution SEM imaging, analytical modeling and finite element simulations. At the slowest strain rates (<0.008/s) microscale glass deforms almost infinitely with a smooth stress-strain behavior; a result of the applied strain being accommodated entirely by the thermally activated shear transformation zones (STZs). At the intermediate strain rates (<0.7/s), the stress-strain behavior is serrated, with each stress-drop corresponding to a respective propagated shear band. Specifically, at a strain rate of ~6/s the microscale glass fails in a purely brittle-like manner, as the speed of shear band propagation is too high to accommodate a large strain. Thus, the deformation of microscale glass at these intermediate strain rates can be attributed to *shear band propagation* kinetics. Interestingly, at even higher strain rates (~60/s to 1335s), the glass micropillars can again sustain significant ductility before failure. The increased plasticity in glass micropillars at these high strain rates will be attributed to the ability of microscale glass to efficiently partition the applied strain to simultaneously nucleated (but not propagated) multiple shear bands aided by the bulk heating of the micropillar. Thus, at such high strain rates, the deformation behavior is controlled by the *shear band nucleation* kinetics.

HIGH RESOLUTION STRAIN-MAPPING DURING IN-SITU NANOINDENTATION OF CVD THIN FILMS

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Key Words: In-situ nanoindentation, scanning X-ray diffraction, time-resolved high-resolution strain-mapping

The NanoMAX beamline is a hard X-ray nanoprobe beamline at MAX IV Laboratory, Lund, Sweden. This beamline was designed to take full advantage of the exceptionally low emittance and the resulting coherence properties of the X-ray beam. A nano-focus beam of 50×50 nm^2 of high X-ray photon intensity is available for experiments. This small focus is ideal to investigate heterogeneous samples in materials science with high spatial resolution, utilizing techniques such as scanning X-ray diffraction, 2D X-ray fluorescence mapping, and coherent imaging in the Bragg geometry.

Chalmers University of Technology and MAX IV Laboratory have acquired a nanoindenter to be installed at the NanoMAX beamline. The combination of *in-situ* micro-mechanical testing and nano-focused scanning X-ray diffraction permits time-resolved high-resolution *in-situ* strain mapping. The experimental configuration is based on an Alemnis nanoindenter which is transferrable between the beamline and a scanning electron microscope (SEM). This allows for a sample characterization in a SEM prior to the X-ray beamline experiment. A potential science case is the investigation of local residual stress fields and their changes under increasing load.

Understanding the deformation mechanisms for thin hard coatings is vital for optimizing their use as wear resistant coatings. This new experimental configuration at NanoMAX will be applied to study the relationship between residual stress state, microstructure, and fracture in self-assembled nanolamellar CVD thin films. We will present the first results of the commissioning of the nanoindenter installed at the NanoMAX beamline at MAX IV and explain potential applications.

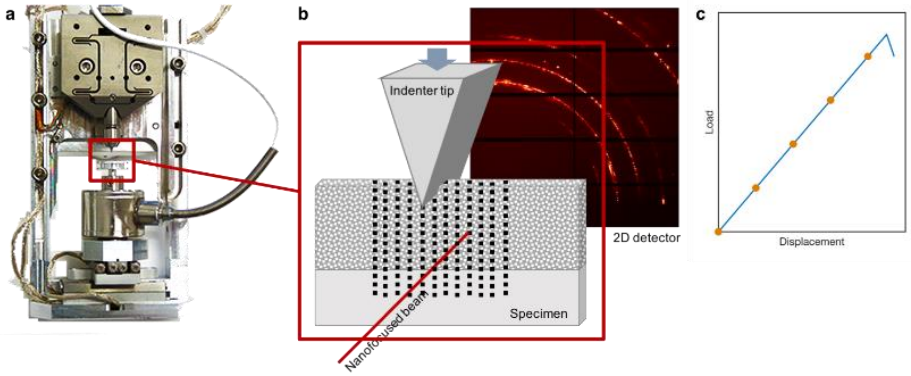

Figure 20. a: Picture of the nanoindenter. The actual size is approximately 65x160 mm. b: Schematic of the wedge indentation process and the mapping of the area below the indenter tip. c: Schematic illustration of a typical loading scheme, where the orange circles mark acquisition of maps for measurement of strain distributions.

CORRELATIVE *IN SITU* TOTAL AND ELASTIC STRAIN MAPPING ON MICROMECHANICAL TESTPIECES BY DIC AND HR-EBSD

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Key Words: Strain mapping, digital image correlation (DIC), high resolution electron backscatter diffraction (HR-EBSD), pre-notched micro-cantilever, micro-tensile

The measurement of elastic strains and stress in micromechanical testpieces upon loading has previously been performed by *in-situ* X-ray synchrotron diffraction and high resolution electron backscatter diffraction (HR-EBSD). Similarly, the measurement of total, elastic and plastic, strains by the technique of digital image correlation (DIC) strain mapping, has recently been performed on micropillars using an applied surface speckle pattern.

In the present study, a method has been developed to combine, correlatively, the techniques of HR-EBSD and DIC to measure *in-situ*, during deformation, the elastic and total strains at the surface of micromechanical testpieces. Hence one may infer the purely plastic component of the total deformation. Practically, this has required the development of a Pt speckle pattern that is not only suitable for DIC, but also does not impede substantially the acquisition of high quality Kikuchi patterns for HR-EBSD.

When applied to the region ahead of a pre-notch in a single crystal W micro-cantilever, stress and strain fields have been measured at sub-100 nm resolution. Furthermore, the direct comparison of the regions of heightened GND content and high plastic strain has revealed that planes of high GND content resulting from deformation are not necessarily planes of high plastic strain. In fact, the prediction of planes of high plastic strain from elastic strain/stress measurements is more subtle. Crack tip toughening mechanisms in metals require the operation of plastic deformation mechanisms, such as slip; this study helps shed light on what local stress states lead to an increased dissipation of mechanical energy. It also confirms the notion that the plastic performance of a material cannot be solely interpreted from elastic strain measurements, even if undertaken *in-situ*, or from post-mortem transmission electron imaging of dislocation structures.

Further discussed examples of the correlative application of these two techniques include microcompression and nano-wedging of Cu, microcompression of lamellar titanium aluminide, and microtensile testing of Mg to determine the local stress state at the onset of twin formation.

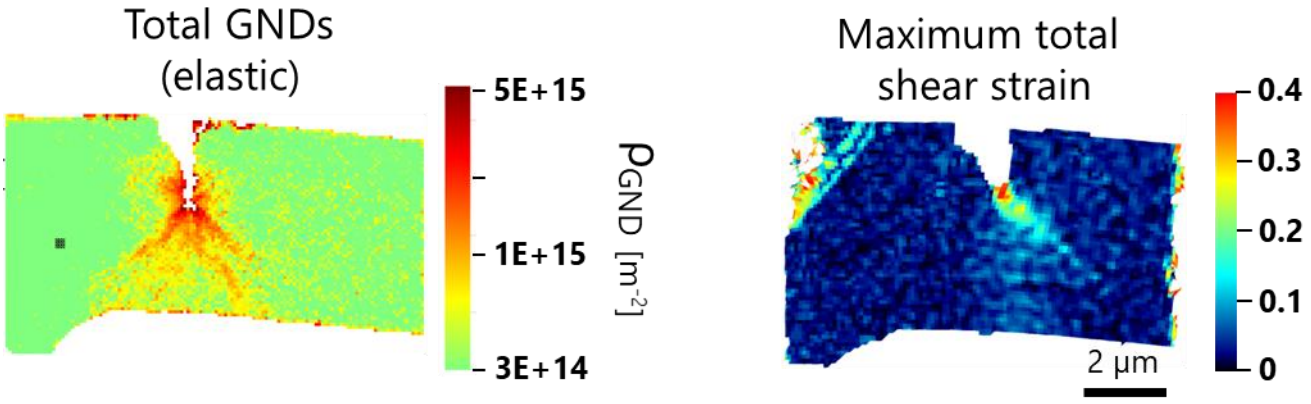

Figure 21 – Left: geometrically necessary dislocation map at the crack tip of a loaded pre-notched W micro-cantilever, measured by HR-EBSD. Right: maximum total shear strain map of the same crack tip, measured by DIC. Note the asymmetry of the total shear strain, compared to the relatively symmetric distribution of GNDs either side of the crack tip.

IN-SITU MICROCOMPRESSION HIGH CYCLE FATIGUE TESTS: UP TO 1KHZ FREQUENCIES AND 10 MILLION OSCILLATION CYCLES

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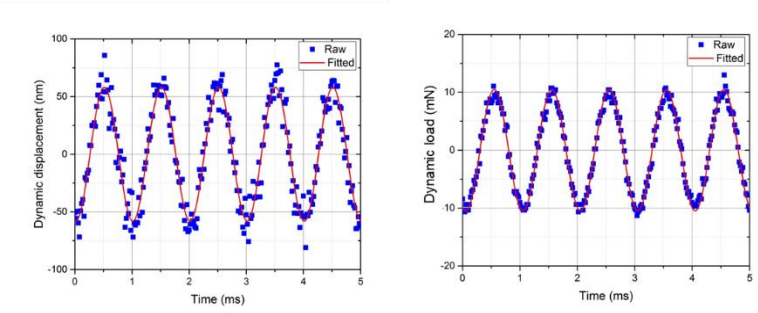
Key Words: Small scale fatigue, high cycle fatigue tests, new technique

Nanomechanical tests are moving beyond hardness and modulus to encompass host of different mechanical properties like strain rate sensitivity [1,2], stress relaxation [3], creep and fracture toughness by taking advantage of focused ion beam milled geometries and well known stress state during testing. Adding high cycle fatigue (HCF) properties to this list will be useful to extend the gamut of properties studied at the micro/nanoscale. There have been several reports of repeated impact and sinus mode (also referred to as “continuous stiffness mode”) nanoindentation tests for studying the contact fatigue properties of films and coatings. Though promising for studying contact fatigue properties, these measurements suffer from low oscillation frequencies (less than ~ 50 Hz) and, consequently, long duration tests. Merle et al. [4] reported micropillar compression-compression fatigue tests on nanocrystalline Cu at 40Hz and required ~ 7 hours to reach 1 million cycles. For a technique like nanoindentation that typically comprises of thermal drift rates of ~ 3nm/min at room temperature, this amounts to a total displacement drift of 1.2µm over the entire duration of the test (7 hours). Therefore, pushing the frequencies of sinus oscillation tests higher seems to be the key towards minimizing artefacts of measurements and to reach high cycle contact fatigue regime in shorter time spans.

This presentation will report the development of micropillar HCF tests with oscillation frequencies up to 1kHz and compression-compression fatigue tests up to 10 million cycles. Micropillar HCF tests performed on single crystal silicon (reference sample, does not exhibit fatigue at high frequencies) showed no change in unloading stiffness over 10 million cycles, suggesting the reliability of the developed experimental technique. The associated instrumentation and technique development, design of the fatigue tests at the micron scale, data analysis methodology, experimental protocol and challenges will be discussed. Compression-compression high cycle micropillar fatigue of nanocrystalline nickel will be presented and the experimental results will be discussed in light of existing literature data, particularly the operative deformation mechanism(s). The fatigue tests were performed both below and above the 0.2% offset yield strength. Prolonged fatigue tests resulted in grain growth and microstructural changes in nanocrystalline nickel. The associated changes in mechanical deformation data (unloading stiffness, load and displacement amplitudes) will be discussed. The convolution of time dependent plasticity in such tests will be addressed by comparing both load and displacement controlled fatigue tests at high frequencies. It is hoped that this study will pave way for routine high cycle fatigue tests of metals at the micron scale and provide clues for designing similar indentation based fatigue tests.



Quality of displacement and load raw data obtained at 1kHz sinus frequency tests on Si micropillars

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SURFACE ACOUSTIC WAVE SPECTROSCOPY VERSUS NANOINDENTATION – POTENTIALS AND LIMITS FOR COATING CHARACTERIZATION

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Key Words: Non-destructive Testing, Surface Acoustic Wave Spectroscopy, Nano Coatings, Quality Control, Elastic Modulus

Measuring mechanical film properties is essential for understanding and designing coating systems as well for controlling quality in coating manufacturing. Since more than two decades, two methods for determining Young's modulus and other mechanical properties of thin films are widely used: The instrumented indentation technique (nanoindentation) and the laser-induced surface acoustic wave spectroscopy (LiSAWS). Due to their different physical principles, both methods address different fields of application but also complement each other. This presentation gives an overview for typical applications, strengths and limits for both methods from practical points of view like precision, requirements for sample material, preparation and test setup, additional results, standardization, and measuring time.

Several examples for the application of both methods are discussed:

- Hard protective coatings (deposited by PVD and thermal spraying) measured with both techniques
- Films with less than 15 nm thickness
- Effect of texture, microstructure and defects on measured properties

MULTI-METAL ELECTROHYDRODYNAMIC REDOX 3D PRINTING AT THE SUBMICRON SCALE: MICROSTRUCTURE – GEOMETRICAL GRADIENTS – CHEMICAL GRADIENTS AND THE RESULTING MECHANICAL PROPERTIES

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Additive manufacturing, metals, gradient materials, nanoscale, microstructure control

An extensive range of metals can be dissolved and re-deposited in liquid solvents using electrochemistry. We harness this concept for additive manufacturing, demonstrating the focused electrohydrodynamic ejection of metal ions dissolved from sacrificial anodes and their subsequent reduction to elemental metals on the substrate. This technique, termed electrohydrodynamic redox printing (EHD-RP), enables the direct, ink-free fabrication of polycrystalline multi-metal 3D structures without the need for post-print processing. On- the-fly switching and mixing of two or more metals printed from a single multichannel nozzle facilitates a chemical feature size of <400 nm with a spatial resolution of 250 nm at printing speeds of up to 10 voxels per second. The additive control of the chemical architecture of materials provided by EHD-RP unlocks the synthesis of 3D bi-metal structures with programmed local properties and opens new avenues for the direct fabrication of chemically architected materials and devices. Mechanical properties can be locally controlled by alloying, dealloying (resulting in controlled porosity) and grain-size tuning via process control. The properties of EHD-RP are put into perspective by comparing with the most prominent current technologies for metal 3D printing at the nanoscale (Fig. 1).

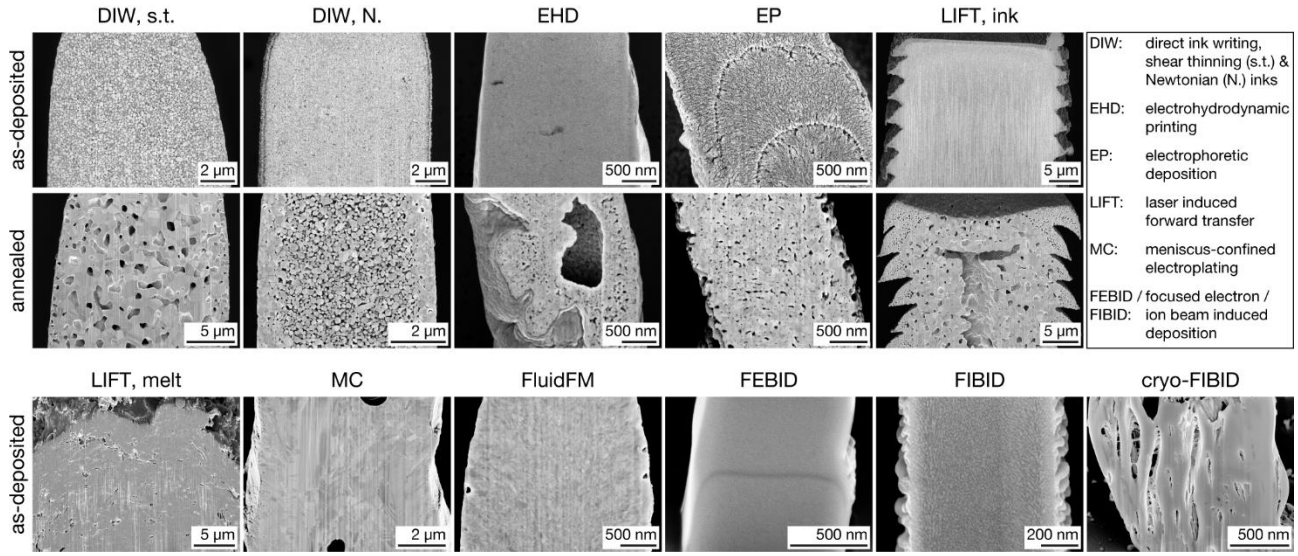

Figure 22 – Comparison of the microstructures of metals deposited by all current processes to 3D print metals at the micron scale

UNDERSTANDING FRACTURE IN LASER ADDITIVE MANUFACTURED BULK METALLIC GLASS THROUGH SMALL-SCALE MECHANICAL MEASUREMENT

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Key Words: *bulk metallic glass; micro-mechanics; additive manufacturing; selective laser melting; fracture*

Bulk metallic glasses (BMGs) are amorphous metal alloys formed by fast cooling that display high strength and toughness with good resistance to corrosion and wear. One traditional limitation has been that BMG castings are often limited to <1 cm dimensions due to the high cooling rates needed. The recent development of selective laser melting (SLM) of metallic glasses opens up the possibility of creating large BMG components with complex geometries. However, we have recently shown that additive manufactured BMGs exhibit poor ductility and toughness when compared to their traditionally as-cast (AC) counterparts (Fig. 1 A-C).

Our work investigates how the processing route influences the structure of a Zr-based BMG, and how this is linked to mechanical performance. Evaluation at the micro-scale is critical, as thermal influences on the microstructure from laser-processing and melt-pool solidification exist at these length-scales. Experimental calorimetry results have shown enthalpic relaxation variation between cast Zr-based glasses and those manufactured with SLM-processing, suggesting differences in free volume for different processing routes. The effect on the fracture properties was studied using single edge notched beam bending tests: SLM-processed alloy showed significantly lower fracture toughness when compared with the as-cast alloy, and this was explained by energetic barriers for activating shear transformations in the glass, elucidated in detail using micro-pillar compression testing (Fig. 1 D/E). These results are further related to the glassy laser-processed structure through advanced structural analyses using synchrotron X-ray diffraction and nanoindentation.

While SLM-processing can effectively overcome critical casting thickness constraints in BMGs, significant issues persist regarding structure and consequently damage tolerance. Our results show that micro- and nano-mechanics is an effective tool for understanding material deformation of laser-processed amorphous alloys at critical length scales. Ongoing research in this area looks to harness small-scale testing as a structural optimisation tool for the development of such advanced alloys.

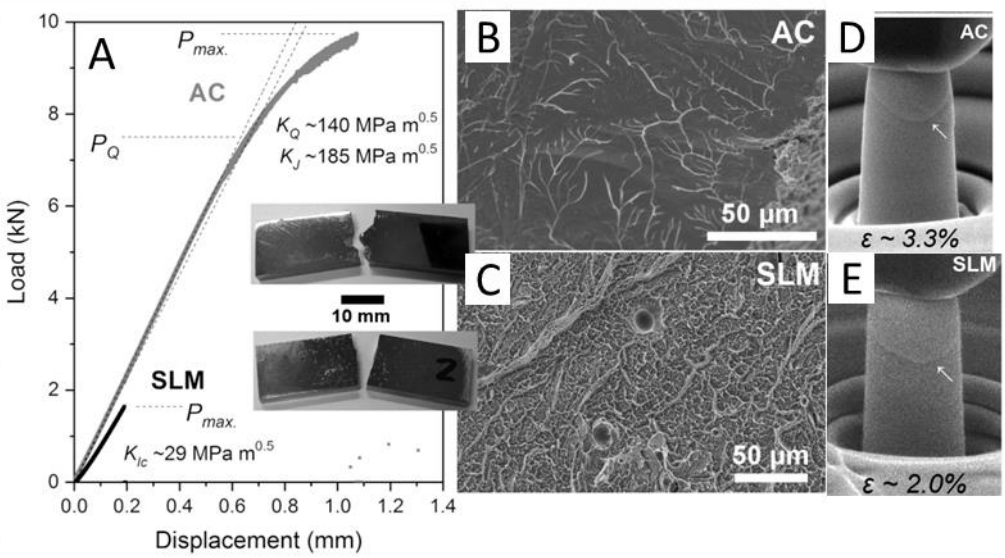

Figure 23 – Macro-scale fracture toughness measurements (A-C) and in situ micro-pillar compression (D/E) of a Zr-based BMG manufactured using casting (AC) and additive manufacturing (SLM).

MICROMECHANICAL TESTING AT HIGH STRAIN RATES AND VARYING TEMPERATURES OF 3D-PRINTED POLYMER STRUCTURES

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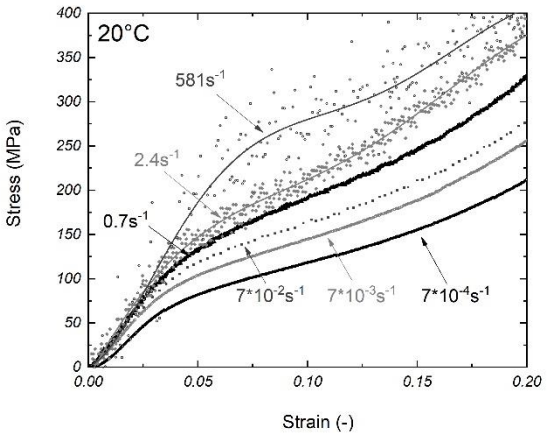
Key Words: Polymer, high strain rate, micro-compression, micro-tensile

Following recent developments, in-situ nanoindenter systems can now perform tests at much faster speeds, enabling us to accomplish micro-compression experiments using strain rates in the range of 10^3 s^{-1} for micron-sized specimens. Thus, it has become possible to study deformation processes occurring in the very small scales of all types of materials. Here, we focus on the effect that dynamic testing speeds have on the mechanical behaviour of 3D-printed micron-sized polymer structures. We look at the influence of temperature and compare results obtained from compression and tensile tests.

All structures were fabricated with a Two-Photon Lithography system using the commercially available photoresist IP-Dip (both by Nanoscribe GmbH). Pillars were printed with a diameter of 6 µm, whereas tensile test specimens had a gauge cross-section of 25 μm^2. The structures were printed onto silica substrates and no further post-processing was done. Micro-compression and tensile tests were conducted using an in-situ nanoindenter from Alemnis GmbH (Thun Switzerland) allowing dynamic and high temperature testing. This way we could apply strain rates spanning seven orders of magnitudes overall with the maximum strain rate accomplished just below 600 s^{-1}.



True stress strain curves determined during micropillar compression tests of polymers using varying strain rates

The stress strain curves obtained in the micro-compression tests at room temperature are given in the figure to the left. The strain rate sensitivity is significant and for a strain rate of 581 s^{-1} yielding only occurred well above 200 MPa, which was roughly 4 times as a high compared to the slowest quasistatic tests. In addition, there was some variation seen in the elastic modulus as well (2.5 to 3.5 GPa), but the increase with strain rate was significantly less pronounced compared to yielding ().

In the experiments conducted here the temperature was increased up to 80°C during testing, which lead to a significant decrease of yield stress and elastic modulus in the quasistatic regime. For higher strain rates, however, the drop in the yield stress was much less pronounced. When comparing the yield extracted from tensile tests to the micro compression results the same strain rate dependency is determined. The values are slightly lower for the yield in the tensile tests, which is likely due to the lack of a hydrostatic pressure component.

To our knowledge this is the first time that small-scale dynamic testing has been performed on polymer micropillars and the development of this experimental technique will be of interest also to the wider micromechanical research community.

SMALL SCALE MECHANICAL TESTING OF NANOPOROUS TUNGSTEN TAILORED BY REVERSE PHASE DISSOLUTION

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Key Words: Nanoporous tungsten, Selective phase dissolution, Microstructure characterization, Mechanical property, Small scale.

Nanoporous metals possess a number of positive attributes such as light weight, large surface area, excellent thermal properties, and energy absorption capability, making them a good candidate as future radiation shielding materials [1]. Tungsten seems to be ideally suited as the base material for such a foam, as it is commonly used in nuclear facilities, medical diagnosis systems and a number of other circumstances in order to protect personnel and sensitive equipment from radiation [2]. Therefore, it is of great value and interest to tailor such novel nanoporous tungsten, in order to combine the beneficial properties of tungsten with the positive attributes of nanoporous foams. In this work, nanoporous tungsten foams with relative densities ranging from 20 to 50 % were created on a bulk scale through a unique route involving severe plastic deformation of a coarse-grained tungsten-copper composite, followed by the selective dissolution of the nobler copper phase. Scanning electron microscopy and high-resolution transmission electron microscopy were utilized for characterizing the microstructural evolution and analyzing the way the etching solutions affect the resulting nanoporous structures. The mechanical properties, which are an important consideration in fusion reactor applications, were investigated by employing nanoindentation and other small-scale testing techniques in situ in the SEM. Based on this, the elemental plasticity mechanisms governing the mechanical behavior were elucidated. This work for the first time provides an innovative and adaptive approach to create bulk nanoporous tungsten. The developed reverse phase dissolution method is generally applicable and can be transferred to other refractory metal materials in the future. The promising mechanical results of nanoporous tungsten will serve as foundation for forthcoming related scientific studies and engineering applications.

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MECHANICAL AND ELECTRICAL FAILURE OF TRANSPARENT NANOWIRE ELECTRODES

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Key Words: five-fold twinned Ag nanowires, transparent flexible electrodes, tensile testing, in situ microscopy

Flexible transparent electrodes have to withstand large mechanical strains without sacrificing electrical performance. For such applications, silver nanowire (Ag NW) networks are highly promising as they combine mechanical flexibility with low sheet resistance and high optical transmittance. In order to improve the performance of such nanowire electrodes a microscopic understanding of the interplay between mechanical and electrical failure is required. This can be achieved by a combination of *in situ* (or interrupted) tensile tests in a scanning electron microscope (SEM) with 4-probe electrical measurements of the sheet resistence.

In the present work the effect of the coating direction on the mechanical and electrical failure of Ag NW electrodes have been studied. For this, the nanowires have been coated in a preferential direction on a thin flexible polymer using doctor-blading. Tensile straining up to 20% plastic strain has been carried out parallel and perpendicular to the preferential coating direction. As can be seen from Fig. 1 (left) the electrical performance shows a strong dependency on the relative orientation of loading and coating direction. Upon straining in preferential coating direction the sheet resistance increases significantly already after 5% plastic strain. In contrast, hardly any increase in sheet resistance is observed upon straining up to 20% perpendicular to the preferential coating direction. This behavior can be understood from SEM images of the Ag NW networks taken at different stages of tensile straining. To illustrate the difference, Fig. 1 shows SEM images of extreme cases where the Ag NW networks are strained to 50%. During straining in preferential coating direction (Fig. 1, middle) most Ag NWs are loaded in tension resulting in periodic (ductile) fracture, which is similar to the behavior of thin metallic films. The corresponding increase in sheet resistance indicates a severe reduction in percolation. In contrast, upon straining perpendicular to the preferential coating direction (Fig. 1, right) only few NWs aligned parallel to the tensile axis rupture whereas the majority of wires oriented perpendicular to the loading direction stay electrically intact. This explains the considerably smaller increase in sheet resistance in this case. Nevertheless, the nanowires aligned perpendicular to the loading direction are also severely plastically deformed. They show frequent kinking (encircled regions) which can be attributed to the lateral contraction of the polymer during tensile straining effectively putting the NWs under compression.

The microscopic structure of the kinks have been studied in detail using high-resolution transmission electron microscopy (HRTEM) which reveals perfect and partial dislocations, high-angle grain boundaries as well as chevron-type defects (not shown). Finally, the microscopic observations are compared with atomistic simulations in order to understand the effect of the five-fold twin structure of the Ag NWs on the bending and kinking behavior. Very good agreement between experiment and simulation is achieved.

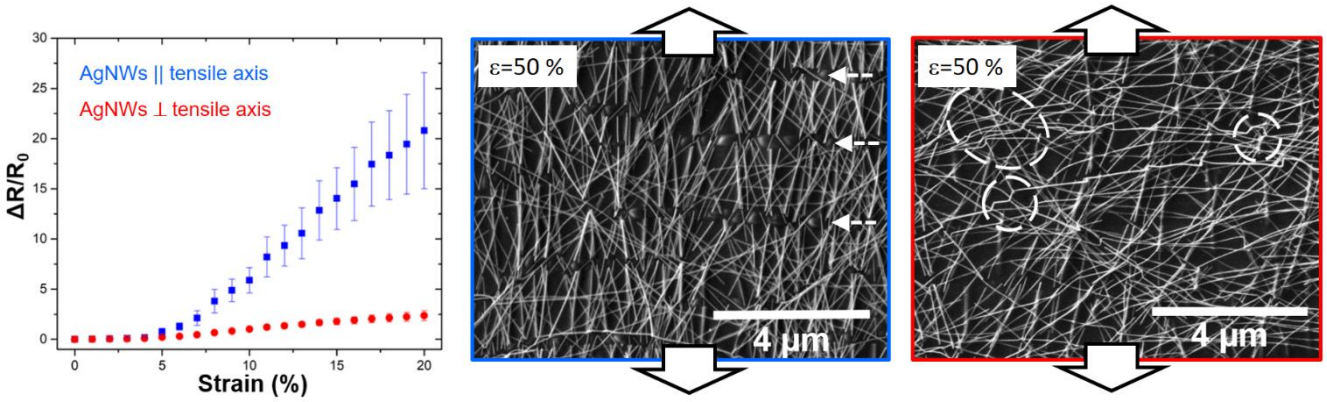

Figure 1 Influence of coating direction on the electrical and mechanical failure of Ag nanowire electrodes during uniaxial tensile testing.

NANOMECHANICAL CHARACTERIZATION OF HIGH PRESSURE TORSION PROCESSED HfNbTaTiZr HIGH ENTROPY ALLOY

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High entropy alloys (HEAs) are a new material class in which the configurational entropy of a multicomponent solid solution phase is maximized so that the entropy of mixing stabilizes disordered solid solution phases against the possible intermetallic phases development. Generally, to achieve high entropy of mixing, the alloys contain typically five or more major elements in equimolar concentrations. The composition of HEAs is generally based on 3d transition metals, refractory metals, light metals, lanthanide transition metals, precious metals, brasses and bronzes. The HEAs exhibit promising structural and mechanical properties in wide range of applications.

Mechanical properties of such alloys can further be improved by grain refinement especially by severe plastic deformation. However, studies of ultrafine grained HEAs are rather scarce in the literature. An increase of strength with decreasing grain size was achieved in the probably most investigated HEA i.e. Cantor alloy (equiatomic CoCrFeMnNi with fcc structure) processed by high pressure torsion (HPT) [1]. Much less attempts were made to process in such a way HEAs with bcc structure. Recently HfNbTaTiZr bcc HEA was successfully nanostructured by HPT straining [2]. It was reported that grain refinement by HPT resulted in a significant enhancement of the strength of this bcc HEA, keeping excellent ductility during room temperature straining. Nevertheless, there is still a lack of information about the development of microstructure and physical properties of this refractory metal HEA subjected to severe plastic deformation processing.

Recent investigations [3] revealed that thermodynamically stable system of HfNbTaTiZr alloy at room temperature is a mechanical mixture of Zr, Hf rich hcp phase and Ta, Nb rich bcc phase. The decomposition of the solid solution after long-term annealing obviously leads to the deterioration of mechanical properties (loss of ductility and decrease of strength). The difference in hardness of both phases is relatively small and both are softer than the random solid solution. On the other hand, considerable contribution to the solid solution strengthening can arise from atomic size misfit (phase separation on the nano-meter scale) which is provoked by the high density of vacancies introduced by HPT.

This work thus aims on the relationship between phase (de)composition, microstructure, lattice defects, and length-scale-dependent material response of HfNbTaTiZr HEA after different thermal treatment and HPT straining. The microstructure and phase composition evolution were characterized by the electron microscopy and X-ray diffraction. The length-scale-dependent material response was characterized by indentation at various indentation depths. The contributions of different hardening mechanisms were separated and attributed to distance between dislocation pinning defects so that the differences between thermal treatment (diffusion) and HPT (straining) -induced hardening could be explained.

Acknowledgement: This research was carried out in frame of the project CZ.02.1.01/0.0/0.0/15_003/0000485 (European Regional Development Fund).

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ELECTROPLASTIC DEFORMATION STUDIES OF AN AL-CU EUTECTIC ALLOY USING NANOINDENTATION

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A promising approach to deform various groups of materials with poor deformability, such as metallic-intermetallic composite materials, is the exploitation of the electroplastic effect, which lowers the yield strength and enhances the elongation to fracture. However, its underlying metal physical phaenomena are not well understood yet.

Since any experimental attempts to further understand the effect have been limited to the macroscopic scale so far, we developed an in-situ electro-nanomechanical testing setup which enables us to apply electric current pulses during indentation. This allows us to electroplastically deform single crystalline phases of defined orientation. Additionally, due to the microscopic contact area, high current densities can be achieved with this setup.

Here, we present our experimental setup as well as recent results on the deformation of the eutectic Al-Al_2Cu system as well as on the single crystalline Al_2Cu phase. These results reveal displacement shifts upon pulsing, with a larger displacement shift following on the first current pulse, indicating that depinning of dislocations from obstacles is the underlying mechanism. Furthermore, a change in shift direction during unloading was observed which is assumed to be caused by long-range internal stress fields present in the deformed microstructure.

CHARACTERIZATION OF PARTICLE DISTRIBUTION IN A BLACK CARBON-FILLED ELASTOMER VIA NANOINDENTATION

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Key Words: Nanoindentation, elastomer, aggregates, heterogeneities.

A new method to characterize the distribution of hard particles dispersed into a soft elastomer matrix is developed using nanoindentation. It is based on the measurement of the contact stiffness from the continuous stiffness measurement module (CSM). Theoretically, for a homogeneous material, the contact stiffness is directly proportional to the contact depth. However, when indenting a carbon black-filled fluoroelastomer (FKM) this relation is no longer valid and abnormal contact stiffness evolutions are measured (jumps).
The tip-particle model developed in this work is simply based on the hypothesis that all the deformation is supported by the elastomer matrix and that black carbon aggregates play the role of hard extensions of the diamond tip, when touching it (grey particles 1,2 & 3, Fig. 1a). As a result, each abnormal variation of contact stiffness is related to a new aggregate in contact with the tip. By knowing the stiffness amplitude of a jump ΔS and the relative stiffness where it appeared S, the equivalent projected area of a particle can be calculated (Fig. 1d). From this calculation, one can extract the distribution of particles surface density from nanoindentation measurements only. Ten experimental indentation tests have been performed and the results are displayed in Fig. 1e. The distribution of particles surface density extracted from experiments is compared to measurements performed by image analysis of a 100 nm thick slide of the material observed by Transmission Electron Microscopy (TEM) (black squares). Furthermore, the tip-particle model is simulated numerically on the same image analysis (down pointing triangles). The results obtained from this model are in excellent agreement with the TEM observation which is really promising. Indeed, this model is an alternative to microscopy characterization which can be complicated to implement.

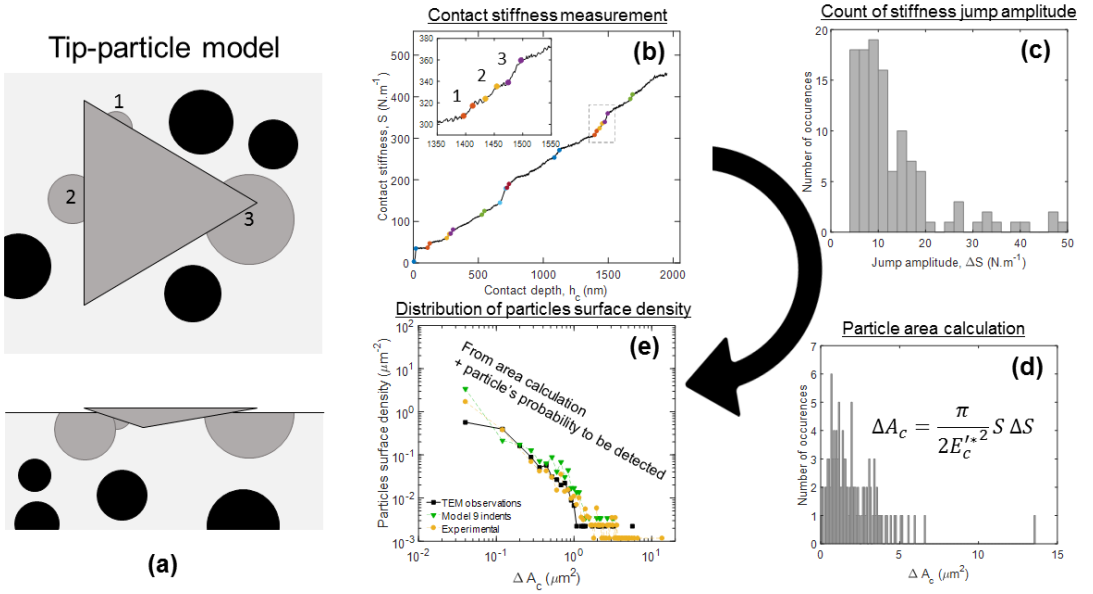

Figure 24 – Basic principle of the tip-particle model applied to a carbon black-filled fluoroelastomer (FKM) : from contact stiffness measurements to distribution of particle surface density as a function of their projected area.