Advanced Characterization Techniques for Coatings and Thin Films

Room Royal Palm 4-6 - Session H2-1

Advanced Mechanical Testing of Surfaces and Coatings

Moderators: Benoit Merle, Friedrich-Alexander-University Erlangen-Nürnberg (FAU), Marco Sebastiani, University of Rome "Roma Tre"

8:00am **H2-1-1 Controlling Disorder in Vapor-deposited Metallic Thin Films and its Influence on Mechanical Behavior***, D Magagnosc,* University of Pennsylvania, USA*; G Balbus,* University of California Santa Barbara, USA*; G Feng,* Villanova University, USA*; **Daniel Gianola,*** University of California Santa Barbara, USA **INVITED**

The nonequilibrium nature of kinetically frozen solids such as metallic glasses (MGs) is at once responsible for their unusual properties, complex and cooperative deformation mechanisms, and their ability to explore various metastable states in the rugged potential energy landscape. These features coupled with the presence of a glass transition temperature, above which the solid flows like a supercooled liquid, open the door to thermoplastic forming operations at low thermal budget as well as thermomechanical treatments that can either age (structurally relax) or rejuvenate the glass. Thus, glasses can exist in various structural states depending on their synthesis method and thermomechanical history. Despite the ability to make MGs in bulk form (cm-size or larger) and their appealing properties, the full spectrum of structural states in MGs and the corresponding mechanical behavior is relatively unknown, stymying the tuning of MG properties via informed processing and synthesis routes. Recent reports of organic glasses synthesized by physical vapor deposition show a degree of control not available in bulk materials and the occurrence of ultrahigh kinetic stability – so-called ultrastable glass formation.

Here, we use sputter deposition while varying the substrate temperature, to isochemically control the structural state and concomitant mechanical response in a Pd-based MG thin film at the time of glass formation. Increasing the deposition temperature from 333 K to 461 K results in a 33.5% increase in hardness to 9.69 GPa for amorphous films. Further increasing the temperature leads to a decrease in hardness, indicating low and high temperature deposition regimes where increased surface mobility allows access to a more relaxed and more rejuvenated structure, respectively. Through this mechanism we access the range of achievable structural states, from ultrastable to highly liquid-like glasses.

8:40am **H2-1-3 Influence of Microstructure on the Cyclic Electro-mechanical Behavior of Ductile Films on Polymer Substrates***, **Megan Cordill,** O Glushko,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences and Monanuniversität Leoben, Austria*; D Többens,* Helmholtz-Zentrum Berlin für Materialien und Energie, Germany*; C Kirchlechner,* Max-Planck-Institut für Eisenforschung GmbH, Germany

In order to advance flexible electronic technologies it is important to study the combined electro-mechanical properties of thin metal films on polymer substrates under mechanical load. Ductile films and lines are an integral part of flexible electronics because they allow current flow between semiconducting islands and other operating features. When ductile films on polymer substrates are strained in tension the substrate can suppress the catastrophic failure that allows for their use in flexible electronics and sensors. However, the charge carrying ductile films must be of an optimum thickness and microstructure for suppression of cracking to occur. In order to improve mechanical and electrical properties of these complex material systems, more work at characterizing the processing-structure-property relationships should be performed. Studies of strained films on polymer substrates tend to emphasize only the electrical properties and thickness effects more than the role of film microstructure or deformation behavior. The microstructure of the film not only determines the mechanical behavior but also influences the electrical behavior and could be optimized if studied in connection with the mechanical behavior. To address both the electro-mechanical and deformation behavior of metal films supported by polymer substrates, in-situ 4 point probe resistance measurements were performed with in-situ confocal scanning laser microscopy imaging of the film surface during cycling. The 4 point probe resistance measurements allow for the examination of the changes in resistance with strain, while the surface imaging permits the visualization of localized thinning and crack formation. Furthermore in-situ synchrotron tensile tests provide information about the stresses in the film and show the yield stress where the deformation initiates and the relaxation of the film during imaging. The combination of electrical measurements, surface imaging, and stress measurements allow for a complete picture of electro-mechanical behavior needed for the improvement and future success of flexible electronic devices.

9:00am **H2-1-4 Crystalline/Amorphous Metallic Multilayers – from Dislocations to Shear Bands***, **Marlene Mühlbacher,*** Montanuniversität Leoben, Austria*; C Gammer,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Austria*; F Spieckermann, C Mitterer, J Eckert,* Montanuniversität Leoben, Austria

Amorphous metallic coatings have recently emerged as promising thin film materials - thin film metallic glasses - due to their excellent chemical stability, good wear resistance and exceptionally high strength. Their mechanical behavior, however, is fundamentally different from their crystalline counterparts, due to the disordered structure lacking dislocations as carriers of plastic deformation. To investigate the different deformation and failure mechanisms, we have synthesized Zr- and Pd-based crystalline/amorphous multilayers with a total thickness below 1 µm and individual layer thicknesses below 100 nm by unbalanced dc magnetron co-sputtering from elemental targets. The microstructural variation is achieved by a change of deposition temperature or a change of composition (e.g. crystalline $Pd_{0.69}Si_{0.31}$ /amorphous $Pd_{0.80}Si_{0.20}$) established by different powers applied to the magnetrons. Nanomechanical samples are prepared in a focused ion beam instrument. Mechanical properties and their dependence on layer thickness and arrangement of the multilayers are investigated with a particular emphasis on *in-situ* tensile testing in the transmission electron microscope. This approach allows for a direct comparison of plastic deformation through the movement of dislocations and shear bands in the crystalline and glassy layers, respectively. Strategies for the prevention of sudden failure of the thin film metallic glass, e.g. by the confinement of shear bands between crystalline layers, are evaluated.

The tensile tests are complemented by *in-situ* and conventional nanoindentation and put into context with glass transition and crystallization temperatures of the thin film metallic glasses obtained by differential scanning calorimetry, thus presenting a comprehensive picture of the crystalline/amorphous multilayer system.

9:20am **H2-1-5 A Novel Method for the Preparation of Tensile Thin Film Specimens for In-situ Mechanical Testing in the TEM***, **Benoit Merle,** J Liebig, M Göken,* Friedrich-Alexander-University Erlangen-Nürnberg (FAU), Germany

A novel method was developed for the preparation of thin film micro-tensile specimens. Unlike most previous techniques, it does neither require the availability of a cleanroom nor that of expensive photolithographic equipment. It is based on a combination of focused ion beam (FIB) milling and electron-beam-assisted etching with xenon difluoride precursor gas. In contrast to existing FIB-based preparation approaches, the area of interest is never exposed to ion beam irradiation and a pristine microstructure is preserved. This is achieved by using a special shadow milling geometry with a thin silicon membrane simultaneously serving as a substrate and protective layer for the thin film of interest. A great advantage of the new method is that it enables the target preparation and mechanical testing of individual microstructural defects. The method was applied to nanotwinned Cu-Al as well as Au thin films. The fabricated tensile specimens were mounted on a push-to-pull conversion device and subsequently tested in-situ in the transmission electron microscope (TEM).

[1] Liebig, J. P., Göken, M., Richter, G., Mačković, M., Przybilla, T., Spiecker, E., Pierron, O.N., Merle, B.: A flexible method for the preparation of thin film samples for in situ TEM characterization combining shadow-FIB milling and electron-beam-assisted etching. Ultramicroscopy, 171:82-88 (2016).

9:40am **H2-1-6 Liquid Metal Embrittlement at the Micro-scale: Gallium FIB vs. Xenon FIB***, Y Xiao,* Laboratory for Nanometallurgy, ETH Zurich, Switzerland*; **Jeff Wheeler,*** Laboratory for Nanometallurgy, ETH Zürich, Switzerland

Micromechanical testing of structures fabricated using focused ion beam (FIB) has allowed significant progress to be made in understand the deformation and properties of small volumes of materials. However, the vast majority of FIB structures are machined using Gallium, which is known to embrittle many metals (e.g. Al, Cu and Fe) by weakening their grain boundaries. This has recently been shown to have a significant negative effect on the strength of micropillars of polycrystalline aluminum. Here we extend upon that work to investigate the effect of Ga FIB on the deformation and fracture properties of grain boundaries of several materials by comparing structures (micro-pillars and -cantilevers) made using both Xe FIB and Ga FIB.

10:00am **H2-1-7 Quantum Contact Mechanics for Tribology, Wear and Erosion*, Norbert Schwarzer,*** SIO, Germany

Erosional or tribological models and simulations do not only require a comprehensive mechanical contact model but also need to account for the principle uncertainties residing in the field.

It will been shown that the classical continuum mechanical and thus, also deterministic, concepts are not adequate if one intends to describe tribological processes like erosion, fretting, wear etc.

By incorporating quantum mechanical concepts via a principle scale dependent accessibility with respect to input parameters from measurement, surface roughness or even non-continuous composition, one does not only overcome such flaws in the classical approaches but also automatically incorporates a method to observe and actively control the influence of the uncertainty budget [1].

According to the classical quantum mechanics, the uncertainty is been accounted for by a “Planck” constant, only that this time, depending on the dimension of the problem, we end up with Planck-vectors or tensors instead of the classical scalar.

The way to go is cumbersome at the beginning, because it requires the principle quantization of the line element of a general continuous space, but the outcome is a very compact, rather general and powerful tool to handle practical applications.

As a byproduct, so it seems, also the quantization of the Einstein field equations can be achieved [2].

[1] N. Schwarzer: „Quantum Tribology – Part I: Theory“, www.amazon.com/dp/ B01CI4BI2E

[2] N. Schwarzer: „Recipe to Quantize the General Theory of Relativity“, www.amazon.com/dp/ B01LX664IF

10:20am **H2-1-8 Textile Nanocharacterization: Topography, Phase Imaging, and Nanomechanical Property Investigation of Polyester Yarn Interaction with Silicon Matrix***, B Kim, **Gerald Pascual**, K Lee,* Park Systems Corporation, USA

Textiles research and development is rapidly turning to nanomaterials to create new fabric blends that have increased performance for traits such as damage resistance, breathability, and even self-cleaning. To better inform materials design strategies, it is necessary to have a tool and techniques capable of measuring not only nanoscale topographies of material components, but their nanomechanical properties as well. Atomic Force Microscopy (AFM) is a solution well-suited to explore and characterize these traits. To this end, a silicon gel matrix and polyester yarn sample was prepared for examination with a commercial AFM system, the Park NX10 from Park Systems. Non-contact mode AFM from Park Systems was used to perform topography and phase imaging. Force-distance spectroscopy plus force-volume mapping was used for nanomechanical property characterization. The acquired data reveals that the hardness of the yarn is about 100 times greater than the matrix it is embedded in with forces being measured in nanonewton resolution and distances in micrometers. This investigation of the textiles sample is reflective of AFM’s effectiveness in allowing textiles researchers to explore the root, nanoscale causes of desirable macroscopic traits in novel fabric blends and further improve upon them.

10:40am **H2-1-9 A Nanoindentation System with Equivalent Capabilities in Both Normal to and Parallel to the Sample Surface*, Warren Oliver,*** Nanomechanics, Inc., USA*; P Phani,* International Advanced Research Centre for Powder Metallurgy & New Materials, India*; K Johanns,* Nanomechanics, Inc., USA*; J Pethica,* CRANN, Trinity College Dublin, Ireland*; K Parks,* Nanomechanics, Inc., USA

An entirely new nanomechanical testing system has been built. The system retains the same measurement capabilities associated with high performance nanoindentation systems from which we have reported results in the past in the direction normal to the surface of the sample and adds the equivalent signals parallel to the surface. The same sensitivity, range and dynamic performance (including frequency specific experiments) are available simultaneously and continuously in both directions. The ability to measure not only load and displacement but stiffness and phase angle at specific frequencies parallel to the surface continuously and simultaneously with these same measurement in the normal direction has resulted in entirely new results concerning the onset of sliding between two bodies in contact. Unique new data concerning the initiation of slip at micro asperities, friction and wear, lubrication, scanning surface topology, mechanical property mapping and multidimensional characterization of structures can now be investigated. Specifics of the dynamic performance when each axis is actuated separately as well as simultaneously will be presented along with a number of examples of its use. A dynamic model that describes the system's behavior will also be presented.

11:00am **H2-1-10 The Effects of TIP Sharpness and Substrate Properties on Nanohardness Measurement in Thin Hard Coatings by FEM*, Frantisek Lofaj,*** *D Nemeth,* Institute of Materials Research of SAS, Slovakia

FEM modelling of nanoindentation in the hard coating/softer substrate system revealed strong influence of the sharpness of the indenter tip on the hardness-indentation depth profiles resulting in the limited validity of the general 10% relative indentation depth rule. The result was attributed to the increase of the size of plastic field under the indenter with the increase of tip radius and the limits for the applicability of the nanoindentation tests with real (blunted) indenters were determined. Another important factor strongly affecting the hardness - depth profiles is the difference among the properties of the coating and substrate. These observations were confirmed experimentally on the corresponding depth profiles obtained in the continuous stiffness measurement mode on different W-C coatings deposited on steel and hardmetal substrates by HIPIMS and HITUS. The above limitations of nanoindentation in the determination of the nanohardness of thin films from the corresponding depth profiles are discussed.

11:20am **H2-1-11 Small Punch Testing for Mechanical Characterisation of a Free-standing CoNiCrAlY Coating*, Hao Chen,*** University of Nottingham, China

In this study, the ductile-to-brittle transition temperature (DBTT) of a high velocity oxy-fuel (HVOF) thermally sprayed CoNiCrAlY (Co-31.7% Ni-20.8% Cr-8.1% Al-0.5% Y (wt%)) coating was investigated. To determine the DBTT, displacement controlled small punch tensile test (SPTT) and multi-step loading small punch test were employed between room temperature (RT) and 750 °C. At low temperatures, evidence of elastic-brittle behaviour was found but at high temperatures extensive yielding and plastic deformation occurred. The yield strength ranged from 1000-1500 MPa below 600 °C to less than 500 MPa above 650 °C and the elastic modulus was found to be approximately 200-230 GPa at 500 °C and 55 GPa above 700 °C, as evaluated via SPTT. The displacements obtained from multi-step loading SPT at each load increment were relatively small and similar at temperatures below 500 °C but a significant increase in displacement was noted at 600 °C. Test results gave a DBTT of this coating of approximately between 500-700 °C. Fractographic investigation showed that the fracture surface at RT exhibited flat, smooth features indicating brittle fracture whereas at 600 °C and above the main fracture mode was dominated by extensive ductile tearing.

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