Advanced Characterization Techniques for Coatings, Thin Films, and Small Volumes
Room Pacific Salon 1 - Session H2-2-MoA
Nanoscale Plasticity

Moderators: Timothy Rupert, University of California, Irvine, USA, **Olivier Pierron**, Georgia Institute of Technology, USA

1:40pm **H2-2-MoA-1 Assessing the Mechanical Properties of Thin Organic Semiconductor Coatings*, Steve Bull,*** Newcastle University, UK

A wide range of organic semiconductor coatings have been developed for optical and electronic applications and have been extensively characterised for their electronic and optical properties. What mechanical measurements have been made are focused on assessing the average properties of a film (e.g. using buckling to assess elastic moduli) but are not suitable to assess point-to-point variation in mechanical response which may be related to changes in coating microstructure due to crystallisation and/or phase separation. The assessment of time-dependent mechanical response is also lacking. This presentation will address the challenges of testing 100-300nm thin films of a range of organic semiconductors on a glass substrate to extract mechanical properties using nanoindentation at very low loads (peak loads less than 50μN). The importance of surface contamination and adhesion of the tip to the film, the effect this has on setting the conditions of first contact and the effect this has on measured properties will be discussed. The use of extrapolation methods to determine coating only properties will be assessed for both quasi-static and dynamic measurement techniques. Finally the effect of coating microstructure and surface roughness on the measured results will be discussed.

2:00pm **H2-2-MoA-2 *In Situ* TEM Activation Volume Measurements*, S Gupta, S Stangebye, J Kacher, Olivier Pierron,*** Georgia Institute of Technology, USA

Signature parameters such as true activation volume are often characterized to identify the governing plastic deformation mechanisms. The current state-of-the-art for characterizing thermally-activated dislocation mechanisms consist of measuring activation volume (using transient tests), along with separate *in situ* TEM observations to provide hints about the actual mechanisms. In this work, we take advantage of recent advances in quantitative *in situ* TEM nanomechanics to simultaneously measure activation volume and perform *in situ* TEM observations of the governing mechanisms. This talk will demonstrate the use of a MEMS device to measure true activation volume based on repeated stress relaxation experiments performed inside the TEM. The technique is demonstrated on 100-nm-thick Au and 200-nm-thick Al micro-specimens, providing true activation volume values of 5 and 10 b^3, respectively. These values will be interpreted in light of the TEM observations performed during these experiments, highlighting mechanisms dominated by grain boundary – dislocation interactions.

2:20pm **H2-2-MoA-3 In-situ Microscale Mechanical Testing of Metal/Ceramic Interfacial Regions*, X Zhang, Y Mu, S Shao, Wen Jin Meng,*** Louisiana State University, USA

Application of ceramic coatings onto substrates is an important means of tuning the near-surface mechanical, chemical, and tribological properties of machining tools and mechanical components for improved performance and durability. Successful application of coatings demands adequate interfacial mechanical integrity [1]. Effective engineering of the coating/substrate interfacial region is predicated on establishing experimental protocols for quantitative measurement of interfacial mechanical response and understanding key mechanisms governing interfacial mechanical failure.

We will summarize our results on using in-situ microscale mechanical testing for quantitatively assessing mechanical integrity of metal/ceramic interfacial regions under shear, compression, and tension loading. Quantitative mechanical testing of interfacial regions was accomplished through in-situ SEM instrumented compression and tension loading of focused ion beam fabricated micro-pillars, in which metal/ceramic interfacial regions are placed at various inclinations with respect to the pillar axis [2,3,4]. Such testing, in combination with in-situ SEM observations and detailed post-mortem characterizations, provides new data and new insights on interfacial mechanical failures under different loading conditions. Accompanying simulations, combining density functional theory, molecular dynamics, and crystal plasticity finite element, provide additional mechanistic interpretations for observed interfacial failures [5,6]. Results of testing on metal/ceramic epitaxial interfaces will also be discussed.

References:

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[4] Y. Mu, X. Zhang, J.W. Hutchinson, W.J. Meng, Measuring critical stress for shear failure of interfacial regions in coating/interlayer/substrate systems through a micro-pillar testing protocol, J. Mater. Res. 32, 1421-1431 (2017).

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2:40pm **H2-2-MoA-4 Nano-wedging: A Novel Test Method to Combine Nanoscale Strain Mapping with Multiaxial Stress States*, Thomas Edwards,*** Empa - Swiss Federal Laboratories for Materials Science and Technology, Switzerland*; F Di Gioacchino, J Pürstl,* University of Cambridge, UK*; X Maeder,* Empa - Swiss Federal Laboratories for Materials Science and Technology, Switzerland*; W Clegg,* University of Cambridge, UK*; J Michler,* Empa - Swiss Federal Laboratories for Materials Science and Technology, Switzerland

A novel small-scale mechanical test geometry is presented, which enables a multi-axial stress state to be generated and the deformation characterised in-situ by two non-destructive strain mapping techniques – digital image correlation (nDIC) of a nanoscale Pt speckle pattern e-beam deposited on the side surface of testpieces, and high resolution electron backscatter diffraction (HR-EBSD). To date, such strain mapping methods have only been applied to small-scale mechanical test geometries where the stress state is nominally uniaxial – micropillars, microcantilevers – or at crack tips. By indenting a vertical wall, supported at both ends, with a wedge-shaped indenter, a stress state can be generated which is similar to that produced by standard surface indentation techniques. This method hence gives insight into the evolution of plastic and elastic strains throughout the depth of the material as indentation progresses, in the loaded or unloaded state. Finite element modelling of the test geometry has enabled the nominal stress distribution to be assessed.

This experimental method has been applied to several pure metals – f.c.c., b.c.c and h.c.p – to investigate the evolution of slip band spacing as a function of indentation depth and local plastic strain. It has also been used on MAX phases investigated as novel coating materials for zirconium-based nuclear reactor fuel rod cladding alloys, where certain deformation mechanisms in the MAX phases are not activated in simple uniaxial straining test geometries.

3:00pm **H2-2-MoA-5 Micromechanical Characterisation of Ag/Au Multilayers by Means of Bulge and Nanoindentation Testing*, Sebastian Krauß,*** *M Göken, B Merle,* Friedrich Alexander-University Erlangen-Nürnberg (FAU), Germany

The mechanical properties of metallic thin films are of paramount importance for the performance and reliability of MEMS devices. An approach to further improve the mechanical properties is to introduce a multilayered structure of alternating materials. Therefore metallic Ag/Au multilayered thin film systems were fabricated by thermal evaporation. The overall thickness of the thin film stacks was kept constant to 800 nm, whereas the number of layers in the film and therefore the individual layer thickness were modified. Multilayered systems containing two to sixteen layers were created, resulting in individual layer thicknesses ranging from 400 nm to 50 nm. The microstructure and morphology of the thin films were investigated by atomic force microscopy (AFM) and by cross-sectioning with a focused ion beam system (FIB). The mechanical properties of the thin films were investigated by nanoindentation on glass substrates and by bulge testing of released membranes. The investigations include hardness, yield stress and fracture toughness analysis and are correlated to the microstructure produced by the evaporation process.

3:20pm **H2-2-MoA-6 Size Effect on Superplastic Flow – In situ Micromechanical Characterization of Superplastic Zn-22% Al**, ***Patrick Feldner***, *M Göken,* University Erlangen-Nürnberg, Germany*; B Merle,* Friedrich Alexander-University Erlangen-Nürnberg (FAU), Germany

Superplastic micro & nanoforming has a great potential for a high throughput production of small-scale structural devices with complex geometries. However, it has not yet been established if the macroscopically observed superplastic behavior also persists at microscopic length scales and which fundamental processes govern structural superplasticity in metallic alloys.

For this reason, the micro & nanomechanical properties of the superplastic alloy Zn-22% Al were characterized as a function of the specimen size, using different, complementary in situ micromechanical testing techniques, including micropillar compression in a scanning electron microscope as well as in a X-ray microscope and tensile testing in a transmission electron microscope.

The resulting deformation kinetics clearly reveal a superplastic trend even at the micro scale. However, below a critical specimen volume a breakdown of the superplastic flow behavior is revealed, which is associated with a loss of ductility. Based on the intra as well as intercrystalline deformation morphology observed during in situ testing, this change of the rate-controlling deformation process is discussed in terms of a transition from boundary mediated ductility to boundary mediated brittleness.

3:40pm **H2-2-MoA-7 Studies on the Mechanisms in Hexagonal Close Packed Metal Nanolaminates**, ***Irene Beyerlein***, University of California, Santa Barbara, USA **INVITED**

The goal of this study is to better understand the mechanisms underlying the mechanical response of nanolayered composites containing either pseudo-morphic body center cubic (BCC) Mg or hexagonal close packed (HCP) Mg phases. Nanolayered composites comprised of 50% volume fraction of Mg and Nb were synthesized using physical vapor deposition with individual layer thicknesses h ranging from 2.5 nm to 50 nm. At the lower layer thicknesses of h < 5 nm the Mg phase was found to have undergone a phase transition from HCP to BCC, such that it formed a coherent interface with the adjoining Nb phase. Hardness testing and micropillar compression testing normal, 45 degrees, and parallel to the interface plane showed that the BCC Mg composite is much stronger and can sustain higher strains to failure. A multiscale, crystal plasticity model incorporating a confined layer slip model for h-dependent critical resolved shear stresses was developed and applied to understand the linkage between the observed deformation response and underlying mechanisms. Calculations from the model predict that the more homogeneous deformation and reduced plastic anisotropy of the bcc Mg/Nb material compared to the hcpMg/Nb results from dislocation-mediated plasticity on the {110} and {112} slip systems in the Mg phase. The hcp Mg/Nb phase exhibits significant plastic anisotropy due to large differences among the slip strengths of the three HCP slip systems.

4:20pm **H2-2-MoA-9 Critical Assessment of the Criteria for Minimum Indentation Spacing**, *S Pardhasaradhi,* ARCI, India*; **Warren Oliver**,* KLA-Tencor, USA

With the advances in nanoindentation measurement instrumentation and the associated testing methodologies, high speed indentation mapping with indents that take less than a second to perform, is now possible. This enables mapping large areas with thousands of indents within a few hours which is extremely useful to measure the local mechanical properties of multi-phase alloys, coatings and small volumes of materials. The combination of high-speed mapping techniques and the continuous push towards understanding the mechanical properties of small volumes of materials, has now put greater emphasis on the minimum spacing of indents. In this work, a critical assessment of the minimum spacing of indents is performed, by a combination of extensive indentation experiments (~ 50000) and finite element simulations at different spacings for a wide range of materials including bulk materials and coatings. It was found that a minimum indent spacing of 10 times the indentation depth is sufficient to obtain accurate results for a Berkovich indenter. This is less than half of the commonly followed criteria of spacing the indents three times the size of the indent, which for a Berkovich indent, is approximately 20 times the indent depth. Similar results were also found for other indenter geometries. Finite element simulations are carried out to visualize the plastic zone beneath the indents and to rationalize the experimental findings. It was found that non-overlapping plastic zones is not a requirement for determining the minimum indent spacing and the observed minimum spacing criteria can be rationalized by simple indentation energy arguments. These results were also found to be applicable for a gold film on a glass substrate, which is an extreme case of soft film on hard substrate that shows significant pile-up. These results have significant ramifications for indentation mapping wherein the indents can now be placed much closer than what was traditionally accepted which enables high resolution mechanical property mapping.

4:40pm **H2-2-MoA-10 Surface Laboratory Assistant – The New Combination of Measurement Device and Analysis Software**, ***Nick Bierwisch***, *N Schwarzer,* SIO, Germany

Nowadays the used materials or material combinations in all application fields (e.g. optical, avionic or automotive industry) are getting more and more complex. These complex structures are needed in order to increase the performance and lifetime of the components. Such improvements of each part of your complex device, tool or structural element are necessary to reach the performance goals demanded by the desired application. This increased complexity demands extended analysis and optimization methods. Classical engineering methods and rules of thumb aren't enough anymore.

Proper characterization and optimization of such structures requires invertable mathematical tools of sufficient holistic character. Unfortunately, as such tools are often unavailable trial and error or half empirical sensitivity analysis methods in combination with FEM or BEM are applied. Thereby faster tools could help significantly to save development time and costs [1].

All models (FEM or analytical based) will need exact and generic material parameters for each part of the material system.

SIO [2] has developed several software modules to determine these parameters from different measurement types and created a variety of easy to use software packages which combine these modules.

There are many different measurement devices, where the results could – in principle – be wonderfully combined and successively be applied to obtain a very holistic picture of the strength and weaknesses of a piece of material one is interested in. Only problem there: So far there was no proper joint sewing all these tests and the results they produce seamlessly together.

This unsatisfying situation has changed now.

The talk will demonstrate how the so-called “Surface-Laboratory-Assistant” automatically not only combines various tests, but also improves them, makes suggestions for the next – higher level – test and even automatically starts and subsequently analyses it.

This way and just by the press of a button and in one go, you could get a most comprehensive and sophisticated holistic material analysis from a simple indentation test, via physical scratch up to complex and application orientated tribo-tests.

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