Advanced Characterization Techniques for Coatings, Thin Films, and Small Volumes
Room Pacific D - Session H2-2-WeM

Advanced Mechanical Testing of Surfaces, Thin Films, Coatings and Small Volumes II

Moderator: Dr. James Gibson, University of Oxford, UK

8:40am **H2-2-WeM-3 The Nature of Defects and their Dynamics Characterized using Scanning Electron Microscopy Approaches***, **Dan S. Gianola**,* University of California Santa Barbara, USA **INVITED**

The past several years has witnessed a surging popularity of two techniques for defect characterization in crystalline materials: (*i*) scanning transmission electron microscopy (STEM) using diffraction contrast imaging, and (*ii*) electron back-scattered diffraction (EBSD) mapping.Here, we link these capabilities by employing a field emission SEM equipped with a transmission detector for defect characterization – termed transmission SEM (TSEM).Imaging modes that are similar to conventional CTEM bright field (BF) and dark field (DF) and STEM are explored, and some of the differences due to the varying accelerating voltages highlighted.We further demonstrate how the richness of information encoded in EBSD patterns is amplified by a new generation of direct electron detectors that enable high speed mapping and acquisition of high-fidelity patterns that can be used for statistically-meaningful defect analyses.Using this new system, we quantify the sharpness of EBSD patterns obtained from several additively manufactured metallic alloys, which reveals sub-grain dislocation structures with high fidelity.Our results demonstrate that the dislocation cell walls produced during fast solidification do not always possess measurable misorientations, and thus do not reflect a geometrically necessary defect organization.Finally, we will show how these techniques can be employed for *in situ* tensile experiments to study the nature of dislocations dynamics in several structural alloys.

9:20am **H2-2-WeM-5 Measurement of Hardness and Elastic Modulus by Depth Sensing Indentation: Improvements to the Technique Based on Continuous Stiffness Measurement***, **Warren Oliver**,* KLA-Tencor, USA*; P. Sudharshan,* ARCI, India*; G. Pharr,* Texas A&M University, USA

The method to measure hardness and elastic modulus of small volumes of material by instrumented indentation presented in the seminal works of Oliver and Pharr in 1992 and 2004, has revolutionized the field of small scale nanomechanical testing. Several recent advances in measurement electronics have enabled testing over a wider range of test conditions (speeds) using methodologies that were developed earlier, which requires a critical assessment. In the backdrop of the latest developments in instrumentation and test methodologies, an overview of the various factors affecting the precision and accuracy of the nanoindentation test results at different test conditions with specific focus on Continuous Stiffness Measurement (CSM) technique will be presented. The CSM technique has also been used to explore the time dependence of material properties.In particular, the stiffness of the contact together with the modulus of the material being characterized gives a direct way of calculating the contact area at any instant that is relatively insensitive to thermally driven displacement drift rates.One of the parameters used to calculate the hardness being measured in such experiments is the load being exerted on the sample by the indenter.The CSM technique requires that the load on the sample be modulated to some degree at a specific frequency.The question arises what value of the load should be used to calculate the hardness when the load is being modulated.Results indicating how the load could be chosen will be presented.

9:40am **H2-2-WeM-6 Ultrasonically Induced Nanofatigue During Nanoindentation***, **Antanas Daugela**,* Nanometronix LLC, USA*; J. Daugela,* Johns Hopkins University, USA

In the era of fast product development thin film developers are looking for quick and efficient methods of characterization.This is especialy true in a semi-conductor industry where advanced multilayered chip/MEMS develpment process needs advanced characterization techniques. Nanoindentation based multi-cycle loading is offering insides into the real-time contact fracture dynamics [1]. A nanofatigue phenomenon can be observed on thin sub-micrometer films by monitoring the resulting multi-cycle nanoindentation loading-unloading curves where post-test imaging helps in identifying materials' behavior [2, 3].In addition, clasical Mason-Coffin and racheting fatigue models derived for the nanoscale contact can be utilized in the predictions and correlate reasonably well with nanofatigue cycles obtained experimentally.

A newly developed ultrasonic nanoindentation tip operates at hundreds of kHz, therefore, inducing milions of load cycles within seconds.The resulting nanofatigue induces different thin film fracture modes such as radial, sink-in and produce unique acoustic signatures.The ultrasonic nanoindentation tip monitors associated waveforms, which can provide additional inside into nanofatigue process dynamics via advanced acoustic waveform analysis.Following our previous study [4], acoustic waveforms were processed using a combination of wavelet based signal decomposition and Deep Learning. The proposed Deep Learning technique yields a reliable classification of acoustic signatures obtained during fracturing ofsub-micrometer thick coatings.

References:

1. B. D. Beake et al, *Materials Science & Engineering A*, 7**80** 139159 (2020)

2. H. Kutomi et al, *Tribology International*, **36**, p.255-259 (2003)

3. Y. Matsuda et al, Wear, **259**, p. 1497–1501 (2005)

4. A Daugela et al, *Materials Science & Engineering A*, **800** 140273 (2021)

11:00am **H2-2-WeM-10 Comparison of Electrical and Image-Based Sensing for Quantitative in Situ TEM Nanomechanical Testing***, S. Stangebye, L. Daza, X. Liu, J. Kacher, **Olivier Pierron**,* Georgia Tech, USA

This presentation describes a microelectromechanical system (MEMS) based, quantitative in situ TEM nanomechanical testing technique to measure the mechanical properties of thin film specimens and characterize their plastic flow kinetics, while observing their deformation mechanisms. The MEMS is comprised of a thermal actuator and load sensor. Two types of sensing techniques, electrical (capacitive) and image-based, are compared in term of their accuracy and precision. The advantages and drawback of each sensing technique are also discussed. The mechanical properties of Al and Au thin films, with a range of thickness (from 25 to 200 nm) and average grain size (from ~50 to 350 nm), and their deformation mechanisms are characterized, and the associated size effects are investigated.

11:20am **H2-2-WeM-11 Understanding the Interface Strain Induced hcp-to-bcc Phase Transformation in Nanolaminate Mg***, K. Jacob,* Iowa State University, USA*; K. Yaddanapudi,* University of California, Davis, USA*; M. Jain,* University of Nevada, Sandia National Laboratory, USA*; J. Michler,* EMPA Swiss Federal Laboratories for Materials Science and Technology, Switzerland*; **Sid Pathak**,* Iowa State University, USA

In this work, we strive to answer fundamental questions related to the hcp-to-bcc pseudomorphic phase transformation of Mg in Nb/Mg nanolaminates. Mg and its alloys attract immense attention for being one of the most promising lightweight structural materials, and are of growing interest to automobile and aircraft industries due to their low density, being 35% lighter than aluminum and 78% lighter than steel . The constituent Mg phase is plastically anisotropic and not ductile due to its inherent hexagonal closed pack (hcp) structure. However, by encouraging a pseudomorphic phase transformation of Mg within the Mg/Nb multilayers, the hcp structure of Mg was transformed to a less anisotropic and more ductile body center cubic (bcc) structure at ambient pressures. The critical layer thickness for stabilizing the pseudomorphic bcc Mg phase (above which the metal reverts back to its traditional hcp structure under ambient conditions) was found to be around 7-8 nm from experimental observations. However this value is significantly larger than the critical layer thickness of 4.2 nm for Mg predicted using an analytical model with density functional theory (DFT) information, or 5 nm from direct thermodynamic calculations. This large discrepancy between experimental and theoretical values clearly indicates that a complete understanding of the underlying mechanisms involved during the phase transformations is still lacking.

This work aims to investigate the following questions: (a) What are the operative mechanisms that control the critical layer thickness of pseudomorphic bcc Mg in a Nb/Mg nanolaminate? In particular, can the discrepancy mentioned above between the experimental and theory/modeling results be explained by considering the effects of the bottom (substrate) vs. top Nb layers separately in a Nb/Mg multilayered structure? (b) What would be the effects of the Nb volume fraction on the resultant structure?

We utilize a novel deposition strategy where bi-layers of Nb/Mg will be terminated/protected by an amorphous coating before further deposition in order to isolate the effects of the bottom (substrate) vs. top Nb layers on the pseudomorphic phase transformation of Mg. We also use micro pillar compression tests to investigate the effects of layer thickness vs. crystal structure (Mg bcc vs. hcp) on these fine-tuned microstructures.

Author Index

Bold page numbers indicate presenter