Thursday Afternoon, November 9, 2023

Advanced Focused Ion Beams Focus Topic
Room A107-109 - Session IB-ThA

In Situ FIB Applications

Moderators: Valerie Brogden, University of Oregon, **Shida Tan**, Intel Corporation

2:20pm **IB-ThA-1 Surface Near Helium Damage in Materials Studied with a High Throughput Implantation Method***, **Peter Hosemann**,* University of California at Berkeley, Lawrence Berkeley National Laboratory*; M. Baloocha, S. Stevenson, Y. Xie,* University of California at Berkeley **INVITED**

Helium damage in materials is of interest to the nuclear fusion, fission and spallation community. Helium generation in bulk material can cause embrittlements and swelling while Helium implantation in surface near areas can lead to blistering, fuzz formation and spalling. All phenomena listed are based on the accumulation of Helium into nanosized bubbles as a function of temperature and external stress states. Studying these phenomena traditionally requires ion beam accelerators and large samples. In this work we introduced nanobeam ion implantation methods which enable rapid multi dose ion beam implantation in surface near regions to enable basic scientific studies in single crystal and polycrystal materials such as Cu, Si, W,. The combination of Helium ion beam implantation using the Helium Ion Beam Microscope, Atomic Force Microscopy, Nanoindentation and Transmission Electron Microscope allows to bring insight into the formation of blisters, the linking up of Helium bubbles and the associated deformation and cracking mechanism. We were able to confirm previously posed hypothesis in tungsten blistering as well as show the dose threshold for silicon amorphization.

3:00pm **IB-ThA-3 Modes of Strain Accommodation in Cu-Nb Multilayered Thin Film on Indentation and Cyclic Shear***, **Mayur Pole**, A. Devaraj, T. Ajantiwalay, S. Tripathi, M. Olszta, T. Wang,* PNNL*; B. Gwalani,* North Carolina State University*; Z. Lu, H. Mehta,* PNNL

Two-phase layered thin films with a high density of semi-coherent interfaces exhibit excellent mechanical properties and thermal stability. In this study, a magnetron-sputtered Cu-Nb dual-layered thin film (~500nm for Cu and ~150nm for Nb) having an amorphous interface between Cu and Nb with a high density of aligned growth twins in Cu is subjected to severe surface deformation. The material is loaded using indentation and cyclic shear under tribological testing. The strain accommodation in the subsurface microstructure after deformation varies based on the local structure and deformation mode. Grain refinement and crack formations in the stressed region of the Nb layer and localized crystallization of the amorphous interface are observed after indentation and scratch testing. Pronounced detwinning of growth twins in the Cu layer under the cyclic shear strain leaves large dislocations sites and loops which are observed both by high-resolution transmission electron microscopy and experiment-guided molecular dynamic (MD) simulations. Our simulations provided insights into understanding the pathway for the detwinning process under cyclic shear loading.

3:20pm **IB-ThA-4 Investigating the Site-Specific Mechanical Properties of Advanced Aluminum Alloys via in-Situ Micromechanical Testing Inside the Plasma FIB***, **Tanvi Ajantiwalay**, A. Devaraj,* Pacific Northwest National Laboratory

Plasma focused ion beam (PFIB) has the potential to fabricate large damage-free specimens for various analytical applications. The use of heavier xenon (Xe) ions instead of conventional gallium (Ga) ions provide faster-milling rates and no ion-implantation. In this work, we demonstrate the utilization of PFIB to fabricate site-specific specimens for micromechanical testing of various aluminum (Al) alloys processed via advanced techniques such as friction stir, and additive manufacturing. Both these techniques modify the local microstructure of the base material to achieve grain refinement and hence optimum mechanical properties. A correlation between the microstructure and mechanical properties is thus established through in situ micromechanical testing inside the PFIB/SEM, which, is eventually beneficial to improve the process parameters and the overall performance of these alloys.

3:40pm **IB-ThA-5 Evolution of Stress Fields During Crack Growth and Arrest in Micro-Cantilevers During *in Situ* Bending***, **Michael Meindlhumer**, M. Alfreider,* Montanuniversität Leoben, Austria*; M. Burghammer, M. Rosenthal,* ESRF, The European Synchrotron, France*; R. Daniel, A. Hohenwarter, C. Mitterer, J. Todt, D. Kiener, J. Keckes,* Montanuniversität Leoben, Austria **INVITED**

In order to improve our understanding of the fracture behaviour in micro-cantilevers it is necessary to elucidate the multiaxial stress and strain fields throughout their irreversible deformation, especially in the regime where simplified homogeneous linear elastic assumptions are not valid anymore In this contribution, cross-sectional X-ray nanodiffraction (CSnanoXRD) with a resolution of 200nm was used for the detection of the multi-axial strain fields associated with crack growth during *in situ*stepwise deformation of (i) a notched clamped cantilever prepared from a multi-layered thin film composed of four alternating brittle CrN and semi-ductile Cr layers on high-speed steel and (ii) a freestanding cantilever fabricated from a nanocrystalline FeCrMnNiCo alloy. Both cantilevers were manufactured by focused ion beam milling. The Cr/CrN clamped cantilever was loaded stepwise to 150 and 460 mN and multi-axial stress distributions were retrieved in a region of interest of 40×30µm^2.

An effective negative stress intensity of −5.9±0.4MPa·m^{½} accompanied by a plastic zone around the notch tip arose in the notched Cr sublayer as a consequence of residual stress in the thin film. The *in situ* experiment indicated a strong influence of the residual stresses on the cross-sectional stress fields evolution and crack arrest capability at the CrN-Cr interface. In detail, crack growth in the notched Cr layer to the adjacent CrN-Cr interface occurred at a critical stress intensity of 2.8±0.5MPa·m^{½}.

The freestanding FeCrMnNiCo cantilever was loaded to 22, 45 and 34mN loads, which corresponds to conditions where elastic loading, crack tip blunting and void formation and coalescence with the crack front are the governing mechanisms, respectively. In that case, CSnanoXRD data were evaluated in a region of 30×35µm^2 centered around the crack tip. At a load of 22mN, a bending stress up to ±1 GPa was evaluated, while directly in front of the notch the crack opening stress raised to 4GPa. In a 200nm circular zone around the notch the measured stress distributions deviated evidently from the linear-elastic fracture mechanics assumptions. At 45mN, crack opening stresses increased to 4.5GPa and a 1µm wide distinct plastic zone formed. Further loading lead to a breakdown of the commonly assumed crack tip singularity and a significant decrease of the evaluated stress magnitude.

The quantitative experimental stress results provide unprecedented insights into the gradual stress evolution at the crack tip and across the cantilevers as well as associated fracture processes in nanocrystalline materials.

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