Advanced Characterization Techniques for Coatings, Thin Films, and Small Volumes
Room Pacific Salon 1 - Session H2-1-MoM
Fatigue and Wear

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

10:00am **H2-1-MoM-1 Acoustic Emission Measurements to Quantifying Damage Accumulation and Crack Initiation in Nickel Single Crystals during High Frequency *In Situ* Cyclic Loading Experiments**, *S Lavenstein, **Jaafar El-Awady**,* Johns Hopkins University, USA **INVITED**

We present a new methodology, coupling between acoustic emission measurements and high frequency *in situ* scanning electron microscopy experiments to quantify the evolution of damage and crack initiation in single crystal nickel microcrystals. The mechanical properties are continuously monitored during the cyclic loading using dynamic measurements and signal analysis. The experimental results show that persistent slip bands (PSBs) form in these microcrystals in the absence of any apparent dislocation dense regions as commonly observed in bulk crystals. In addition, the speed of propagation of these PSBs is measured. Quantification of crack initiation and propagation is also analyzed from acoustic emission measurements, and the statistics of these measurements at different stages of crack propagation (i.e. short crack and long crack regimes) are quantified.

10:40am **H2-1-MoM-3 A Data-driven Approach to Describe Fatigue Damage Evolution and Crack Initiation in a BCC Steel Microstructure**, *A Durmaz, **Thomas Straub**, C Eberl,* Fraunhofer IWM, Germany

A material's fatigue lifetime is determined by the crack formation process: damage accumulation in individual grains, micro crack initiation, and finally short crack formation. In the past years, a testing methodology for fatigue damage evolution investigation was developed. This methodology reduced samples sizes and cycled them at their resonant frequency, using sensitive measurements of the resonant frequency for correlation with damage initiation.

Building on this work, a multi modal approach has been developed employing in-situ optical images of the sample surface to better understand damage evolution kinetics and improve crack initiation analysis. The processed image data allows the creation of labels for machine learning (ML) methods, such as random forests, which are based on decision trees. As ML attributes various microstructure characteristics are extracted for each grain using complementing ex-situ EBSD measurements and MTEX. In order to generate a sufficient and balanced dataset the SEM and EBSD methods were automated.

11:00am **H2-1-MoM-4 Low and High Cycle Fatigue Testing of Ni Microbeams**, ***Alejandro Barrios,*** Georgia Institute of Technology, USA*; E Kakandar,* Cranfield University, UK*; X Maeder,* Empa - Swiss Federal Laboratories for Materials Science and Technology, Switzerland*; G Castelluccio,* Cranfield University, UK*; O Pierron,* Georgia Institute of Technology, USA

Small-scale fatigue is an active area of research due to the widespread use of metallic films and micrometer-scale structures in applications such as flexible electronics, and micro electromechanical systems (MEMS). New techniques are required to characterize the fatigue damage and its size effects in metallic microcomponents under loading conditions relevant to their applications. This work presents two small-scale fatigue testing techniques to characterize the fatigue behavior of electroplated Ni microbeams subjected to in-situ high cycle and low cycle fatigue loading conditions. The in-situ high cycle fatigue technique consists of MEMS microresonators that are driven at resonance inside a Scanning Electron Microscope (SEM), leading to fully-reversed loading of microbeams at a frequency of ~8 kHz. The fatigue damage leads to a decrease of the microresonator's resonance frequency and is measured to quantify crack growth rates. The low cycle fatigue technique consists of the external mechanical actuation of the microresonator using a micromanipulator, which exerts a close to fully reversed bending fatigue loading on the microbeam. The micromanipulator is attached to a load cell and the decrease in load needed to actuate the device as cycling increases is used to characterize fatigue damage. Both techniques were complemented with Focused Ion Beam (FIB) cross sectioning that allow for a better understanding of the mechanisms of crack nucleation and propagation.

The crack propagation rates on the surface of the microbeam in the high cycle regime are extremely low (average values down to ~10^−14 m/cycle) indicating that the fatigue mechanism in high cycle fatigue does not follow the common crack tip stress intensification. Instead, crack nucleation and propagation are caused by the formation of voids that nucleate from the condensation of vacancies. However, in the low cycle fatigue regime, the microbeam does follow the conventional fatigue mechanisms observed in literature. Results will highlight the comparison in fatigue life and mechanisms of the low cycle and high cycle fatigue regimes. In addition, further testing will evaluate the frequency effects by comparing low cycle and high cycle tests at similar stress amplitudes. Electron backscatter diffraction (EBSD) scans will also allow for a better understanding of the microstructural variation along the crack path in the microbeam.

Furthermore, modeling efforts with 3D crystal plasticity will complement experimental results by giving a more complete understanding of the stress/strain states at crack initiation sites and a prediction of the low cycle fatigue life of the microbeam at various loadings.

11:20am **H2-1-MoM-5 Nanocrystalline Alloys with Disordered Complexions Probed by In Situ Mechanical Testing**, ***Timothy Rupert,** J Wardini, J Schuler,* University of California, Irvine, USA

Recent innovations in materials processing have enabled the creation of nanostructured materials with unique grain boundary structures. Here, we focus on nanocrystalline metals with amorphous intergranular films, which have been predicted to add a toughening effect. Due to the limited volumes of materials that can be made on lab scales or the geometry of typical parts, it is difficult to accurately probe the mechanical properties of these materials. In this talk, we first describe the use of in situ mechanical testing in the scanning electron microscope, with the goal of measuring important properties only from the regions of interest. We focus on properties of fundamental importance, such as yield strength, strain hardening rate, ductility, and rate sensitivity, with measurements made by microtension and microcompression of very small samples. In addition, in situ fatigue testing inside of the transmission electron microscope provides an atomic scale view of plasticity near a developing crack in materials with and without the amorphous films. Using these results, we revisit the design of these materials, to suggest paths for improvement in the future.

11:40am **H2-1-MoM-6 Structural Evolution and Wear-rate Transitions in Nanocrystalline Alloys**, ***Olivia Donaldson,** J Panzarino, T Rupert,* University of California, Irvine, USA

Nanocrystalline alloys have shown great potential as wear resistant coatings due to their high strength and hardness, but cyclic plastic deformation associated with sliding contact can lead to grain coarsening. In this study, we explore near-surface microstructural changes resulting from scratch wear tests in a nanoindenter, with a focus on understanding how such evolution affects subsequent wear properties. Electrodeposited Ni-W films, with an initial grain size of 3 nm, underwent transmission electron microscopy characterization of the grain structure and texture following scratch wear under normal loads of 10 to 50 mN. Additional Ni-W films with a grain size of 45 nm, achieved through annealing, underwent wear testing as well to provide a contrasting example where damage appears as grain refinement. A clear connection between instantaneous wear rate and subsurface microstructure was found. In addition, the final grain size for the damage layer near the surface was observed to be strongly dependent on the applied normal force, suggesting that local stresses near the surface affect the metastable, near-surface grain size that forms during wear. Finally, a wear map which captures microstructural changes due to different experimental testing variables was constructed and preliminary experiments on Cu-rich alloys were performed to provide a comparison.

12:00pm **H2-1-MoM-7 Effects of Thermal Cycling on Nano-mechanical Properties of Thermal Barrier Coatings**, ***Marco Sebastiani,*** Roma TRE University, Italy

In the present work, we analysed the nano-mechanical properties of high- and low-porosity Yttria-partially stabilized zirconia – YSZ top coats and of the thermally grown oxide (TGO) layer in a thermal barrier coating (TBC), produced by thermal spray.

High-speed nanoindentation and micro-pillar splitting were used for spatially-resolved analysis of elastic modulus, hardness and fracture toughness of the materials as a function of the number of thermal cycles.

In this way, the degradation of both elastic modulus and hardness of the YSZ top-coat is quantified, and a correlation with observed microstructures is proposed.

In addition, the micro-scale fracture toughness of the TGO layer is measured, for the very first time, as a function of the number of thermal cycles.

The existence of a critical TGO thickness of ≈5 µm, postulated by previous research, is confirmed.

Until this threshold, the TGO is solely based on aluminium oxide, it grows slowly and generally remains dense and compact, except for some prominent asperities where the TGO becomes thicker. As a result, its micro-scale fracture toughness of the TGO also tends to increase up to maxima of 2.5 – 3 MPa·√m.

These findings have implications on the failure mechanisms. Indeed, in addition to large stress concentrations along the interface, it is inferred that a reduction in the properties of both the TGO and the top coat contribute to failure of the TBC.

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