Advanced Characterization Techniques for Coatings, Thin Films, and Small Volumes
Room Pacific D - Session H3-TuA

Characterization of Coatings and Small Volumes in Harsh Environments

Moderators: Thomas Edwards, Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland, **Peter Hosemann**, University of California, Berkeley, USA

2:20pm **H3-TuA-3 Stabilized Nanocrystalline Thin Films for Enhanced Thermal, Radiation, and Mechanical Performance***, **Brad Boyce (blboyce@sandia.gov)**,* Sandia National Laboratories, USA **INVITED**

Nanocrystalline thin films can be alloyed to promote thermal stability via solute segregation to grain boundaries.The segregation can impede boundary migration kinetically via solute drag or Zener pinning, or thermodynamically by reducing the boundary's energetic cost.In this study we examine a Pt-Au alloy where the Au has been shown to promote boundary stability upon annealing.Going beyond the thermal contribution, we explore the Pt-Au alloys performance under monotonic tension, fatigue loading, wear loading, and ion irradiation.In each of these cases, the presence of solute can provide synergistic benefits on material properties.However, there are cases where the solute is also detrimental; for example, an over abundance of Au at the grain boundaries can lead to an overall embrittlement and reduced ductility.Through a series of in-situ TEM, in-situ SEM, and ex-situ experiments, we explore the complex role of solute segregation on the thermal, mechanical, and radiation properties.Finally, we propose a chemical pathway to achieve gradient nanostructured metals via compositional tailoring.Unlike the existing plastic deformation methods, like surface mechanical attrition, the compositional tailoring can lead to a heterogeneous grain structure that is 'antifragile': naturally stabilized against further evolution.

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3:00pm **H3-TuA-5 Explaining How *H/E* Influences Coating System Wear Under Harsh Conditions - Insights from Elevated Temperature Nanoindentation, Scratch and Impact Tests***, **Ben Beake (ben@micromaterials.co.uk)**,* Micro Materials Ltd, UK

The ratio of hardness to elastic modulus (H/E) of coating systems and their wear resistance under harsh conditions has been explored. Small-scale tribo-testing at room and elevated temperature (to 600C) has been used to simplify the wear conditions, allowing the role of contact severity, length scale and damage tolerance to be studied to determine why coating optimisation strategies are effective and why they can fail.

Results show the importance of relatively low elastic modulus in reducing tensile stresses in sliding/abrasive contact. This is a key factor in why coating design for optimised H/E and resistance to plastic deformation, H3/E2, can be more effective than aiming for extremely high hardness.

The influence of substrate ductility and load support on the damage tolerance of the coating system in impact tests has been investigated by testing at different contact size (nano/micro/macro). The combination of a coating with moderate hardness, high plasticity index and a tough (i.e. damage tolerant) substrate can improve impact resistance.

The results of the small scale tests show that mechanical and microstructural factors should not be considered in isolation. The role of coating microstructural design in optimising the high temperature mechanical properties of coating systems and their performance in tribologically severe contact conditions such as high speed machining is highlighted.

4:00pm **H3-TuA-8 *In-Sem* Micromechanical Testing Up to 1000 °C of High Entropy Transition Metal Nitride Thin Films Alloyed With Al***, A. Pshyk,* Linköping University, IFM, Sweden*; **Thomas Edwards (thomas.edwards@empa.ch)**,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland*; B. Bakhit,* Linköping University, IFM, Sweden*; M. Jain,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland*; P. Küttel,* Alemnis AG, Switzerland*; G. Greczynski, L. Hultmann,* Linköping University, IFM, Sweden*; J. Michler,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland

Transition metal nitride (TMN) based thin films have a hugely diverse range of applications due to their unique properties including high hardness, thermal stability, high-temperature oxidation resistance and low coefficient of friction. Recently, high entropy transition metal nitrides (HENs) have proven to be among the most promising ways to advance TMN-based thin films. HENs have gained considerable interest in the scientific community, demonstrating a blend of properties often highly enhanced in comparison to their conventional TMN counterparts. Although the outstanding properties of HENs have been well demonstrated at room temperature, their thermal stability and related high temperature mechanical properties are rarely investigated: the question of their potential as high temperature structural materials remains open.

Here, we investigated the mechanical properties of different HENs at temperatures up to 1000 °C by micromechanical testing. Equimolar (TiHfNbVZr)N and (TiHfNbVZrTa)N thin films alloyed with different Al content were deposited using a novel hybrid method combining high-power impulse magnetron sputtering (HiPIMS) from an Al target with DC magnetron sputtering (DCMS) from high entropy alloy targets, i.e. equiatomic TiHfNbVZr and TiHfNbVZrTa targets, in which a negative substrate bias is synchronized with the Al-rich portion of the HiPIMS pulse. The elevated temperature performance of the HENs was evaluated using a state-of-the-art high-temperature micromechanical testing system to carry out micropillar compression. The tests were performed *in situ* in an SEM at 25, 500, 700, 800, 900 and 1000 °C under vacuum, allowing observation of the deformation mechanism changes at elevated temperature, and avoiding oxidation. This direct measurement of mechanical properties (strength, plasticity limit, stress-strain behaviour, brittle-to-ductile transition) of HENs at elevated temperatures was interpreted in relation to the microstructure, phase composition, lattice distortion and valance electron concentration of the HENs, as well as phase changes upon annealing, e.g. spinodal decomposition. Further high temperature notched microcantilever fracture toughness measurements are currently underway.

Experimental details such as indenter material selection and chemical reactivity are also discussed.

4:20pm **H3-TuA-9 Custom Cryo-Nanoindenter for in-Situ Investigations of the Brittle-to-Ductile Transition in a Scanning Electron Microscope***, **Hendrik Holz (Hendrik.Holz@fau.de)**, S. Gabel, B. Merle,* University Erlangen-Nuernberg, Germany

Hydrogen is a promising energy carrier, which however needs to be transported and stored at cryogenic temperatures. A more thorough understanding of the mechanical behaviour of structural materials and coatings at those low temperatures is crucial to ensure safe operations. While nanomechanical testing of materials at elevated temperatures has gained traction over the past few years, only a handful of nanoindentation systems are yet commercially available for operation below room temperature. In this work, we present a novel custom-built cryo-cooled in-situ nanoindenter, which is operated inside a scanning electron microscope. This nanoindenter is based on a commercial system from Femtotools, where we have added low-vibration gas flow cooling and temperature control. In this presentation, we will show first applications to the brittle-to-ductile transition of chromium. Chromium is commonly used as a coating on top of steel and is therefore a natural candidate material for liquid hydrogen tanks. Its suitability for cryogenic application is paramount.

4:40pm **H3-TuA-10 Development of a Novel High Strain Rate Nanoindenter for Small-Scale Mechanical Characterization Over a Wide Strain Rate Range***, **Stefan Zeiler (stefan.sz.zeiler@fau.de)**, H. Holz, B. Merle,* University Erlangen-Nuernberg, Germany

Understanding the changes in mechanical behavior at high deformation speeds and the influence of the microstructure are crucial steps to increase the damage tolerance of components that are exposed to impacts during their lifetime, e.g. safety-relevant components in cars and airplanes. Currently, there is a deficit in experimental techniques for probing the mechanical behavior of coatings and small volumes at high strain rates. In this presentation, we will introduce a nanoindenter with enhanced electronic components, which was used to overcome the indentation strain rate limitation of conventional nanoindentation (ca. 0.1 s^{-1}). An intrinsically displacement-controlled piezo-actuator is operated in combination with a piezo-based load cell and a 1 MHz data acquisition system. Novel testing methods allow measurements with constant indentation strain rates up to ca. 10^4 s^{-1}. This presentation will focus on challenges with the experimental procedures and show first applications to superplastic alloys over several orders of magnitude of indentation strain rate.

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