Friday Morning, April 27, 2018

Advanced Characterization Techniques for Coatings and Thin Films
Room Royal Palm 1-3 - Session H3

Characterization of Coatings in Harsh Environments

Moderators: JeffreyM. Wheeler, ETH Zürich, James Gibson, RWTH Aachen University

8:00am **H3-1 Zr/Nb Nano-multilayers – Structural and Mechanical Response to Radiation Damage***, M Callisti,* University of Cambridge, UK*;* ***Tomas Polcar****,* University of Southampton, UK

Zr/Nb nanoscale metallic multilayers (NMMs) with a periodicity (*L*) in the range 6 – 167 nm were prepared by magnetron sputtering studied by a combination of transmission electron microscopy analyses and nanomechanical measurements to reveal deformation and strengthening mechanisms. Electron diffraction analyses revealed a change in the crystallographic orientation of α-Zr when *L* 27 nm, while Nb structure retained the same orientations regardless of *L*. For $L > 60$ nm, the strengthening mechanism is well described by the Hall-Petch model, while for $27 < L < 60$ nm the refined CLS model comes into picture. A decrease in strength is found for $L < 27$ nm; plastic strain measured across compressed NMMs revealed a change in the plastic behaviour of α-Zr, which experienced a hard-to-soft transition. Multilayers were subject to high energy implantation (He, C, Si, Cu), gamma and proton irradiation, and the effects of radiation damage on mechanical properties were studied in detail. DFT simulations were used to identify helium diffusion and agglomeration in pristine and radiation-damaged Zr/Nb interfaces.

8:20am **H3-2 Nanoindentation of Commercial PVD Hard Coatings at Elevated Temperatures***, W Oliver,* Nanomechanics, Inc., USA*; M Romach,* Advanced Coating Service (ACS), USA*; R Anthony,* ***Kurt Johanns****,* Nanomechanics, Inc., USA

Ten different commercial PVD hard coatings have been characterized with nanoindentation experiments. The thickness of each film has been measured along with a pass/fail test for adhesion. The results from polished surfaces and the rougher as deposited surfaces have been compared. High-speed (NanoBlitz) experiments and statistical analysis have been used to understand the relationships between polished surface and as deposited results. The number of tests required to properly characterize the as deposited surface has been determined. In addition, a selection of the samples have been characterized with high speed, high temperature experiments. The hardness and modulus distributions at room temperature and high temperatures will be presented.

8:40am **H3-3 Elevated Temperature Micro-impact Testing of TiAlSiN Coatings***,* ***Ben Beake****, A Bird,* Micro Materials Ltd, UK*; L Arrom,* Cranfield University, UK*; F Jiang,* Huaqiao University, China

In developing advanced wear-resistant coatings for tribologically extreme highly loaded applications such as high speed metal cutting a critical requirement is to investigate their behaviour at elevated temperature since the cutting process generates frictional heat which can raise the temperature in the cutting zone to 700 °C or more. It has been shown previously that high temperature nanomechanical characterisation can be a valuable tool in understanding coating properties and how they will perform in different types of cutting tests with different requirements [1]. High temperature micro-tribological tests, such as micro-scratch and nano-impact extend the characterisation capability and have provided severe tests for coatings and correlate well to cutting tool life [2].

In this study we report the development of a novel micro-scale elevated temperature impact capability capable of producing repetitive impacts at significantly higher strain rate and energy than in the nano-impact test. With the new experimental test capability it is possible to study coating fatigue with less sharp spherical indenters than in the nano-impact test.

Micro-impact, nanoindentation and micro-scratch tests have been performed to 600 ºC on monolayer and nanolaminate TiAlSiN coatings on WC-Co. The key role of the elevated temperature nanomechanical properties in the impact behaviour of the coatings is discussed and correlations to elevated temperature micro-scratch tests to 600 ºC are investigated.

[1] *Progress in high temperature nanomechanical testing of coatings for optimising their performance in high speed machining*, B.D. Beake and G.S. Fox-Rabinovich, Surf. Coat. Technol. 255 (2014) 102.

[2] *Elevated temperature repetitive micro-scratch testing of AlCrN, TiAlN and AlTiN PVD coatings*, B.D. Beake, J.L. Endrino, C. Kimpton, G.S. Fox-Rabinovich and S.C. Veldhuis, International Journal of Refractory Metals and Hard Materials (2017).

9:00am **H3-4 Fracture Testing of Transition Metal (Oxy)Nitride Coatings***,* ***James Gibson****, S Rezaei, H Rueß, M Hans, D Music, O Hunold, S Wulfinghoff, J Schneider, S Reese, S Korte-Kerzel,* RWTH Aachen University, Germany

Transition metal (oxy)nitride coatings are used in polymer forming operations for a combination of outstanding wear resistance and chemical compatibility with the polymer materials. Varying the chemical composition and deposition parameters for the coatings will optimise mechanical properties by a combination of chemistry and microstructural optimisation. By developing a representative model for these materials, these materials can be rapidly and efficiently prototyped and improved. However, as both chemistry and microstructure play a role in the material properties, both of these variables must be taken account of in this model. This work demonstrates the first steps in linking quantum-mechanics, micro-mechanics, and meso-scale finite element models together in order to fully understand the behaviour of these coatings.

The effect of thin film composition and temperature on the elastic, plastic and fracture properties of transition metal nitride and oxynitride coatings was invested by nanoindentation, micro-cantilever bending and micro-pillar compression. Vanadium and titanium aluminium nitride and oxynitride coatings were manufactured by high-power impulse magnetron sputtering on silicon substrates. A focused ion beam was used to cut notched micro-cantilever beams to determine values of fracture toughness and micro-pillars were cut to try and obtain plastic deformation in otherwise brittle coatings. Tests were carried out to 500˚C in-situ using a Nanomechanics inSEM system. Results are explained via DFT modelling of the coating chemistry, and integrated into a cohesive-zone element finite element model.

9:20am **H3-5 In-situ Study of Deformation and Fracture Processes in Nanostructured Metals at Elevated Temperatures***,* ***Daniel Kiener****,* Montanuniversität Leoben, Austria **INVITED**

Understanding the mechanical deformation and failure processes that take place in nanostructured bulk materials or for thin films on substrates is of prime importance for further improvement of the related material performance.

Miniaturized experiments provide the possibility to specifically test thin layers, individual phases, or interfaces, thereby extending our material understanding. In this presentation, recent developments using quantitative in-situ deformation testing and in-situ fracture experiments within scanning and transmission electron microscopes at ambient and elevated temperatures will be presented. The detailed observations accessible by such advanced experimental setups will be addressed in several case studies concerning the deformation and fracture behavior of, for example, nanostructured bcc metals and layered structures.

10:00am **H3-7 Cryogenic Micropillar Compression Transient Tests at the Lower Limit of Crystallinity Case Study: Nanocrystalline Palladium-Gold***,* ***Juri Wehrs****,* Platit AG, Switzerland*; J Schwiedrzik,* EMPA - Swiss Federal Laboratories for Materials Science and Technology, Switzerland*; M Deckarm,* Universität des Saarlandes, Germany*; J Wheeler,* ETH Zürich, Switzerland*; X Maeder,* EMPA - Swiss Federal Laboratories for Materials Science and Technology, Switzerland*; R Birringer,* Universität des Saarlandes, Germany*; J Michler,* EMPA - Swiss Federal Laboratories for Materials Science and Technology, Switzerland

The plasticity of nanocrystalline metals is governed by a complex ensemble of deformation mechanisms which strongly depends on the materials grain size. Smaller grains are less effective in generating dislocations and hence their ability to interact across intercrystalline domains is reduced. Therefore it is instructive that, in particular for that case that grain sizes approach the limit of crystallinity towards the amorphous regime, grain boundary-mediated deformation processes gain influence while dislocation-mediated processes fade. Mechanisms which essentially emerge from the core regions of grain boundaries, such as grain boundary sliding, grain boundary migration, dislocation nucleation and shear transformation zones are under debate. Consequently, both thermally activated and inelastic, stress-driven deformation processes can be simultaneously operative in these materials. All of these mechanisms contribute towards the increased time dependent plasticity of nanocrystalline metals, manifesting itself as a high degree of strain-rate sensitivity and susceptibility to load relaxation and creep even at room temperature.

In this study we explore the strain rate sensitivity of a highly pure nanocrystalline $Pd^{90}Au^{10}$ alloy with an extremely fine nominal grain size of d~10nm by means of dynamic micropillar compression experiments in a temperature interval from -55°C to 250°C. First we introduce and discuss the novel testing technique, our experimental considerations and data analysis methods. Then we focus on the applicability of this type of micromechanical experiment for probing activation parameters in nanocrystalline materials. The extracted activation parameters (i.e. strain rate sensitivity, activation volume and activation energy) are discussed and compared to literature data to gain insights into the possible rate controlling deformation mechanisms at the lower limit of crystallinity.

10:20am **H3-8 Surface Roughness Effects of Hard Coatings under Three-body Abrasive Sliding Conditions***, **Reza Gheisari**, A Polycarpou,* Texas A&M University, USA

Three-body abrasive laboratory experiments were conducted using two different hard coatings intended for abrasive conditions for electrical submersible pumps (ESP) used in the oil and gas industry. The coatings used were specifically made for the above-mentioned application namely chromium carbide, and Diamonize coatings. These coatings have different surface topographies as well as different mechanical properties. Abrasive slurry of laboratory size-controlled silica sand in hydraulic oil was used to simulate the operational lubrication condition for the ESP. A specialized tribometer was used that simulated temperature, pressure, and velocity conditions encountered in ESPs and uses a pin-on-disk configuration, submerged in the slurry. In–situ measurements of the normal and friction forces were performed. A contact pressure of 6 MPa at a sliding velocity of 1.92 m/s was applied on the contact for 3×10^4 cycles, corresponding to 3500 meters. The focus of the study was to investigate the synergistic roles of the mechanical and roughness properties of the coatings on the wear and friction under three-body abrasive conditions. SEM and EDS analyses on the surfaces, as well as surface profilometry of the wear tracks were employed to gain more detailed perspective of the tribological mechanisms active at the macro and micro levels. It was concluded that while the effect of hardness ratio of the coating to the abrasive is righteously highlighted in previous studies, an RMS roughness close to 1 µm could significantly enhance the wear and friction performance of the coatings. In addition the ratio of abrasive size to surface roughness was proposed as an important factor to be taken into consideration while selecting the optimized coating for an abrasive condition.

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