Tribology and Mechanical Behavior of Coatings and Engineered Surfaces

Room San Diego - Session E2-2-TuA

Mechanical Properties and Adhesion II

Moderators: Megan J. Cordill, Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben, **Ming-Tzer Lin**, National Chung Hsing University & Chaoyang University of Technology

2:00pm **E2-2-TuA-2 Mechanical Behavior Study of 50 nm-thick Thin Film of Gold Single Crystal with In situ X-ray Pole Figures Measurements**, ***Pierre-Olivier Renault,*** Université de Poitiers, France*; J Drieu La Rochelle, P Godard, M Drouet, J Nicolai, M Beaufort,* University of Poitiers, France*; D Thiaudière, C Mocuta,* SOLEIL Synchrotron, France

For a few years, thanks to the development of mechanical tests on nanometer-size objects, the question of how the mechanical plastic behavior of metallic materials change as their characteristic sizes are reduced down to nanometer range has attracted significant interest. In addition to slip by dislocation glide, deformation twinning is one of the plastic deformation mechanisms in metallic crystalline materials. In this way, size-dependent twin propagation behaviors in nanowires such as face-centered cubic metals has been reported in literature.

In this work, the mechanical behavior of gold single crystal is studied thanks to in situ pole figures measurements during controlled biaxial loadings. The gold thin film 50 nm thick is deposited on NaCl single crystal by physical vapor deposition technique at 400°C. The film is then transfered on a polyimide cruciform substrate to be deformed on a biaxial tensile tester in situ during synchrotron x-ray measurements. The as-deposited gold single crystal contains a given amount of small twins. The twins are rather narrow and small, i.e. they have a thickness of about 10 nm and a size of about 50-100 nm.

In the presentation, the first results obtained on in situ uniaxial applied deformation in the [110] direction of Au monocrystalline film are reported. The in situ x-ray pole figure measurements show a huge evolution of the intensity of the diffracting pole related to the twins. Hence, the amount of twins is increasing during deformation, either by thickening or growing of the pre-existing twins, either by creating new twins. The twins' volume is quantified as a function of applied deformation.

2:20pm **E2-2-TuA-3 Evaluation of the Mechanical Properties in Antibacterial Multi-layer HA-Ag Coatings Deposited by RF Magnetron Sputtering**, ***Julian Lenis,*** *M Gómez, F Bolívar,* University of Antioquia, Colombia

The use of osteoconductive coatings such as Hydroxyapatite (HA) in surgical implants is a great alternative in order to improve the acceptance of these devices in the human body. On the other hand, in order to avoid possible problems associated with bacterial infections, which can be generated during the surgical or postoperative procedure, researchers have been developing antibacterial coatings that act locally inhibiting the bio-film formation. One of the strategies that has been adopted to induce these properties is the incorporation of silver (Ag) nano-particles in HA coatings. In the literature it has been found that these coatings exhibit a good antibacterial behavior, however, there are few studies about the mechanical behavior of these systems deposited like multi-layer coatings. Therefore, in the present report the effect of the process parameters such as deposit temperature and substrate-target distance, on the chemical composition, structure, morphology and mechanical properties of multilayer coatings HA-Ag deposited by magnetron sputtering on Ti6Al4V is shown. The characterization of the obtained films was carried out by means of energy dispersive spectroscopy, micro-Raman spectroscopy, scanning electron microscopy, atomic force microscopy and nano indentation tests. A relationship between the target-substrate distance and the chemical composition of the coatings was found, obtaining a decrease in the Ca/P ratio, as function of the decrease between the target and substrates. Additionally, the critical load of the deposited coatings shown an increasing tendency as function of the decrease in the target-substrate distance, whereas for the first level of cohesive failure an inverse behavior was obtained. Furthermore, both mechanical characteristics were improved with the increase of the deposit temperature.

Key words: magnetron sputtering, hydroxyapatite, multi-layer coating, critical load, first level of cohesive failure.

2:40pm **E2-2-TuA-4 Mechanical Deformation in Metal and Ceramic Nano Multilayers**, ***Andrea Hodge,*** University of Southern California, USA **INVITED**

Nano multilayers (NMs) consist of alternating layers of materials with thicknesses on the order of nanometers and typically display many attractive properties which are attributed to the fact that, as the layer thicknesses decrease, the individual layer behavior changes and the interface volume increases. In general, studies on the mechanical behavior of NMs have been focused mostly on metal systems, followed by metal/ceramic systems and even fewer studies on ceramic multilayers. Thus, the behavior of metallic NMs is well characterized and general trends such as a Hall-Petch type strengthening behavior has been observed for variety of material systems as result of the layered structure. In contrast, for ceramic NMs, the research is limited and overall trends and mechanisms have not been well determined. Furthermore, for NM ceramics, the role of dislocations is limited and therefore the role of the interfaces increase and hold the key to understanding their mechanical behavior.

In this study, we present a comprehensive microstructural evaluation of metal and ceramic multilayers with various layer thicknesses and compositions in order to elucidate on the role of their interfaces during mechanical deformation. Several NM configurations including SiO_2/TiO_2, AlN/SiO_2, AlN/Ag, Cu/Nb, Mo/Au and Hf/Ti will be presented. The role of bilayer thickness and composition is evaluated in both compression and tension using nanoindentation and micro-tensile tests.

4:00pm **E2-2-TuA-8 Deposition of Highly Adhesive Ta Based Thin Films on a Biomedical Grade CoCrMo Alloy**, ***Jesus Corona-Gomez,*** *Q Yang, Y Li,* University of Saskatchewan, Canada

Tantalum (Ta) based thin films including alpha and beta tantalum, TaN, and Ta doped diamond-like carbon (Ta-DLC) thin films deposited by radio frequency (RF) magnetron sputtering were investigated as potential interlayers to enhance DLC coating adhesion on a biomedical grade CoCrMo alloy. The deposited interlayers and DLC thin films were characterized by X-ray diffraction, Raman spectroscopy, and Rockwell C indentation. The correlation between the adhesion and the structure of interlayers was investigated. The results show that the deposition temperature and bias voltage play an important role in determining the structure and adhesion of the interlayers and thus the adhesion of DLC coatings.

4:20pm **E2-2-TuA-9 DIC on FIB Ring-Core of Thin Films for Depth Sensing Residual Stress Measurement**, ***Ming-Tzer Lin,*** *W Pan,* National Chung Hsing University, Taiwan*; T Chen, F Cheng,* National Cheng Kung University, Taiwan*; J Huang,* National Tsing Hua University, Taiwan

Reliable measurement and modeling of residual stresses at the micrometer scale is a great challenging task for small-scale structures and nanostructured thin films. Moreover, the specific location on microscale evaluation of residual stress gradients is a very critical issue in the hard coating of thin films. The analysis of the residual strain depth profiles requires detailed knowledge of the in-depth lattice strain function, so the residual stress profile calculation can be carried out in a manner that takes into account the mechanical anisotropy and texture of the materials. The development of a microstructure independent procedure for depth-resolved measurement of residual stress is an issue of strategic interest. Here, we perform a digital correlation (DIC) of the specimen images acquired by incremental focused ion beam (FIB) ring-core drilling with various depth steps. A translation test was performed first to study the applicability of DIC to FIB images, and a proper procedure was established to obtain more accurate results. Next, 2 um thick sputtered Ag and ZrN thin films were used for this measurement. To observe the depth-resolved residual stress profiles of each step on thin film samples, two FIB images of the specimen, one before and one after being drilled, were processed to extract the surface deformation from tiny changes in the FIB images using DIC. This combined with high-resolution in situ SEM imaging of the relaxing surface and a full field strain analysis by digital image correlation (DIC). A parallel residual stress measurement was also performed using a four-circle diffractometer with grazing incidence X-ray diffraction (XRD) $\cos^2\alpha\sin^2\Psi$ method at several azimuthal angles to obtain the average X-ray strain (AXS) and thus the residual stress can be accurately determined and compared.

4:40pm **E2-2-TuA-10 Metallic Glass/Crystalline Nanolayered Coatings with High Nanoscratch Resistance and Damage Tolerance**, *M Abboud,* Middle East Technical University, Turkey*; A Motallebzadeh,* Koç University, Turkey*; **Sezer Özerinç,*** Middle East Technical University, Turkey

Metallic glass/crystalline metal nanolayers provide a balanced combination of high hardness and ductility, making them promising materials for wear resistant coating applications. However, there has been no study to date investigating the wear behavior of these materials. In this study, we investigated the nanoscratch behavior of nanolayered CuZr/Zr composed of alternating CuZr metallic glass and nanocrystalline Zr layers.

A magnetron sputterer deposited the 1 µm thick coatings on polished 316 stainless steel substrates. Layer thickness varied in the range 10-100 nm. X-ray diffraction and transmission electron microscopy characterized the microstructure of the coatings and nanoindentation measurements probed the hardness and elastic modulus. A nanoindenter equipped with a sphero-conical diamond tip of 5 µm radius performed nanoscratch measurements on the coatings.

Hardness and elastic modulus of nanolayered coatings do not vary with layer thickness and are close to that of the monolithic metallic glass, about 5.5 GPa, and 105 GPa, respectively. A comparison of nanoscratch residual depths, on the other hand, indicate that monolithic CuZr has higher scratch resistance than the layered samples. Among the nanolayers, CuZr/Zr with 100 nm layer thickness shows the highest scratch resistance, and the resistance diminishes with decreasing layer thickness. All coatings showed considerably smaller penetration depth when compared to uncoated stainless steel demonstrating the superior wear resistance and damage tolerance of the coatings.

A close observation of scratch tracks showed outstanding adhesion and conformity of the coatings to the substrates. The most damage tolerant coating was the CuZr/Zr with 10 nm layers, showing no sign of delamination or fracture at compressive strain levels exceeding 80%. Monolithic CuZr showed local delamination and microfracture at relatively lower strain levels, but without any gross failure.

Although the hardness and elastic modulus of the coatings are virtually the same, the nanoscratch results indicated distinct patterns. We attribute the layer-thickness dependent behavior to the low interfacial shear strength in these layers previously reported in literature. While low interfacial strength does not play a major role in nanoindentation response where plastic strain is mostly perpendicular to the interface, it affects the scratch resistance where shear strain is dominant. Our findings give insight to the behavior of metallic glass/crystalline composites under sliding loading conditions for the first time in the literature, and present a model system for the development of protective coatings suitable for compliant substrates.

5:00pm **E2-2-TuA-11 Coatings Effect On Crack Initiation Behavior Of Ti Alloys**, ***Xiaolu Pang,*** University of Science and Technology Beijing, China

Titanium has high strength/weight ratio, stiffness and corrosion properties, but at the same time different kinds of surface treatments are employed due to the poor wear resistance, and surface coating is one of the most popular ones. Since fatigue is one of the most important properties of titanium alloys and fatigue crack initiation period occupy an important position in the fatigue failure process, so the study on coating effect on fatigue crack initiation should play an important role. However, the effect of coatings on fatigue crack initiation of Ti-alloys has not be taken seriously, and the mechanism is not clear. This paper deposited CrAlN and TiN hard coatings by PVD on the surface of TC4 titanium alloy to study the coating effects on fatigue crack initiation and propagation. Hard coatings significantly decreased fatigue properties of TC4, since the 510-530 MPa TC4 fatigue limit was reduced to 315-330 MPa for the CrAlN coated samples. The existence of coatings prevented deformation of the TC4 samples at the beginning of fatigue tests, and had a positive effect on sample deformation in the middle of the fatigue process. For the coated samples, the fatigue crack initiated in the coatings and propagated to the interface, and induced a micro crack at the substrate surface. Then the micro crack propagated into the bulk material and formed the distinct propagation path in the fracture surface. The mechanism of non-propagation fatigue cracks and influence of coating thickness were also studied. A corresponding model is proposed.

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