Advanced Characterization Techniques for Coatings, Thin Films, and Small Volumes
Room Pacific D - Session H2-1-TuM

Advanced Mechanical Testing of Surfaces, Thin Films, Coatings and Small Volumes I
Moderator: Prof. Olivier Pierron, Georgia Institute of Technology, USA

8:40am **H2-1-TuM-3 Multifunctional Characterization of Nanomultilayers*, Andrea Maria Hodge,*** University of Southern California, USA **INVITED**

Nano multilayers (NMs) films 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 this presentation, I will discuss how to synthesize and characterize systems of nanostructured multilayers, leveraging nanoscale features to enhance properties and function. To address this, samples with a wide range of composition and layer thicknesses were synthesized via DC/RF and reactive magnetron sputtering. Multilayer configurations of metal/metal, ceramic/metal and ceramic/ceramic systems were designed as model systems for either optical optimization or thermal studies. A comprehensive microstructural and mechanical evaluation of selected metal and ceramic multilayers are presented in order to elucidate on the role of their interfaces for properties and function.Several NMs configurations including SiO_2/TiO_2, AlN/SiO_2, AlN/Ag, Fe/W, and Mo/Au be discussed. The role of bilayer thickness and composition are evaluated and related to final microstructure and behavior.

9:20am **H2-1-TuM-5 Effects of Radiation Damage on the Critical Resolved Shear Stresses in Zirconium Alloys for Nuclear Applications*, James Gibson,*** *C. Grovenor, A. Wilkinson,* Oxford University, UK

Nuclear power provides 10% of the world’s electricity and is likely to expand as countries seek to provide low-carbon energy in the future. Of the current operating 442 commercial nuclear reactors, 96% are water cooled and thus have their uranium oxide fuel contained within a zirconium alloy cladding. This cladding limits fission product release into the primary water loop, as well as acting as the main transport medium for neutrons and heat.

Under irradiation, like most metals, these zirconium alloys exhibit strong hardening effects. Typically, the yield stress of Zr-alloys doubles during the early stage of irradiation while a dramatic loss of strain-to-failure is observed. Irradiation hardening is currently qualitatively described by <a> loops being barriers for dislocations during mechanical loading. This hardening is also supplemented by irradiation-induced precipitates, but their effect on irradiation hardening in Zr-alloys is currently unknown.

Like most of other hexagonal close packed (HCP) materials, zirconium deforms anisotropically via plastic slip on the basal, prismatic and pyramidal planes. As loop formation is also crystallographically influenced, a full picture of the radiation damage effects in zirconium must be gathered on a piece-by-piece basis, with the influence of damage levels, strain rate and temperature being determined on each slip system.

We present here the first steps towards painting this picture of radiation damage effects in zirconium. Nano-indentation testing using spherical and Berkovich tips correlated with indented grain orientations from EBSD has been used for initial rapid screening of irradiation hardening and strain softening effects, as required by complementary CP-FEM models. Subsequently identified “interesting” samples have been selected for more quantitative micro-mechanical investigation to determine critical resolved shear stresses on specific slip systems.

9:40am **H2-1-TuM-6 Link between Cracking Mechanisms of Trilayer Films on Flexible Substrates and Electro-Mechanical Reliability Under Biaxial Loading*, Shuhel Altaf Husain,*** Institut Pprime - CNRS - ENSMA - Université de Poitiers, France*; P. Kreiml,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben, Austria*; P. Renault,* Institut Pprime - CNRS - ENSMA - Université de Poitiers, France*; C. Mitterer,* Montanuniversität Leoben, Leoben, Austria*; M. Cordill,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben, Austria*; D. Faurie,* CNRS, France

Flexible electronics is a technological innovation that involves the use of flexible polymer substrates as the basis structure for the assembly of electronic circuits [1–3]. Currently, flexible electronic devices have new emerging applications, especially for foldable displays and even in biology for integration on skin [4,5]. Flexible polymeric substrates show the advantages of low cost, light weight, mechanical compliance and bendability [6]. Nonetheless, these systems suffer from limited durability, both mechanically and electrically due to the multi-cracking phenomenon during loading [7]. Many works have studied the mechanical behavior of multilayers in order to profit from interfaces and mechanical contrasts (ductile/brittle, stiffness) between each layer to improve the durability [8].

In this work, the propagation of cracks from a top layer in trilayer systems (Cr/Cu/Mo) on a polyimide substrate is studied experimentally by in situ synchrotron X-ray diffraction under equi-biaxial loading (see figure 1). The results show that depending on the thickness of the ductile Cu middle layer (100 nm or 500 nm), the propagation can be a direct vertical path through all layers or a more complex path. These effects are analyzed by monitoring the individual stresses of each layer along with electrical resistance and resulting crack patterns. Cracks starting from the upper Cr layer propagate instantaneously through the whole system for a 100 nm Cu layer, but are strongly deflected in a 500 nm Cu layer, thus delaying the global fracture of the system measured by the increase of electrical resistance. Mechanisms are proposed and allow to anticipate the electro-mechanical performances of stretchable systems constructed of several layers.

[1] S. Yuvaraja, A. Nawaz, Q. Liu, D. Dubal, S.G. Surya, K.N. Salama, P. Sonar, Chem. Soc. Rev. 49 (2020) 3423–3460.

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[3] H. Li, Q. Zhan, Y. Liu, L. Liu, H. Yang, Z. Zuo, T. Shang, B. Wang, R.W. Li, ACS Nano 10 (2016) 4403–4409.

[4] D. Makarov, Nat. Mater. 20 (2021) 1589–1590.

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10:00am **H2-1-TuM-7 Effect of Nanometric Stacking on the Magneto-Mechanical Properties of Thin Films on Flexible Substrate***, H. Ben Mahmoud,* ***Damien Faurie,*** CNRS-LSPM, France*; P. Renault,* CNRS-Pprime, France*; F. Zighem,* CNRS-LSPM, France

Flexible or stretchable magnetic systems are of growing interest in the scientific community, on the one hand for the fundamental aspects of magnetomechanical couplings in low dimensional objects and on the other hand for the numerous applications they can generate (magnetoelectric deformable devices for media, textiles or the human body, etc). Thus, it is important for the community to acquire numerical and experimental means to better understand the evolution of the physical properties (magnetic and electrical) of these flexible systems when they are subjected to deformations [1-2].

Regarding the experimental means, it is essential to be able to evaluate the evolution of the magnetization curves at strain levels comparable to those of a real use (a few ten of percent). Indeed, these strains applied at the macroscopic scale can have repercussions at the microscopic level on the organization of the material (in this case magnetic) and affect the functional properties. In the case of nanometer-thick films on deformable substrates (such as polymers), this can in most cases be characterized by cracks that multiply during the mechanical test. This multi-cracking phenomenon can be complex and involves, by the creation of new surfaces, a strong heterogeneity of stress (and strain) at the scale of the inter-crack fragments, but with a rather weak evolution of the stress averaged on the whole thin film.

However, the links between these mechanical phenomena and magnetic properties have been little explored. Currently, the most ambitious in situ developments have been either at small strains (piezoactuation or bending, up to 1-2%), or at large strains for the properties of giant magnetoresistance. Actually, there is a lack of measurements of magnetization curves at large strains.

In this presentation, we present in situ magneto-mechanical experiments carried on 50 nm Co and NiFe thin films deposited on a polyimide substrate in order to highlight links between damaging of these nanometric films and their magnetic properties. Moreover we will show that the addition of weak layer (W) in the systems affects on the one hand the pattern of cracks and consequently the magnetomechanical properties (evolution of coercive and saturation field, magnetic anisotropy and remanent magnetization).

[1] F. Zighem & D. Faurie, « A review on nanostructured thin films on flexible substrates: links between strains and magnetic properties », Journal of Physics: Condensed Matter 33, 233002 (2021)

[2] D. Faurie, A. O. Adeyeye, F. Zighem, « Prospects towards stretchable and flexible magnonic systems», Journal of Applied Physics 130, 150901 (2021)

10:20am **H2-1-TuM-8 Influence of the Aspect Ratio of the Micro-Cantilever on the Determined Young's Modulus Using the Euler-Bernoulli Equation***, F. Konstantiniuk,* Montanuniversität Leoben, Austria*; M. Krobath, W. Ecker,* Materials Center Leoben Forschungs GmbH, Austria*; C. Czettl,* CERATIZIT Austria GmbH, Austria*; **Nina Schalk**,* Montanuniversität Leoben, Austria*; M. Tkadletz,* Montanuniversitat Leoben, Austria

Micro-cantilever bending experiments can be used to determine fundamental material properties, such as fracture stress and fracture toughness. Furthermore, the Young's modulus can be calculated from the slope of the load-displacement curve using the classical Euler-Bernoulli equation. However, in literature it can be frequently found that applying this technique, the Young's modulus is significantly underestimated, especially when using micro-cantilevers with aspect ratios (bending length/width) < 6. In order to investigate the influence of the aspect ratio on the determination of the Young's modulus, SiO_2 and single crystalline α-Al_2O_3 micro-cantilevers with aspect ratios ranging from ~3.5 to 6.5 were fabricated using focused ion beam milling and subsequently tested with a nanoindenter system. In addition to the quasi-static tests, dynamic micro-cantilever bending tests were performed by using an excitation frequency on top of a quasi-static loading and the results of both techniques were compared to each other. Finally, finite element analysis was used to simulate the static-micro-cantilever bending experiments and investigate the influence of shear deformation, flexing of the support and the geometry of the micro-cantilever on the load-displacement curves.

10:40am **H2-1-TuM-9 Engineering Metal-MAX Phase Multilayered Nanolaminates for Tunable Strength and Toughness***, **Skye Supakul**, S. Pathak,* Iowa State University, USA*; K. Yaddanapudi,* University of California at Davis, USA

Metal-Ceramic nanocomposites have long been of interest to combine the high strength of ceramics with the toughness in metals. To achieve this combination, metal-ceramic multilayers have relied on significantly reduced bilayer thickness (few nm's) and crystalline components, leveraging improved plastic co-deformation for the improved mechanical properties. Here, we discuss the process of designing metal-MAX phase multilayered nanolaminates (MMN) which leverages the unique combination of metallic, covalent, and ionic bonding in MAX phases to enhance the threshold thickness for improved plastic co-deformation and enhanced toughness. Furthermore, with the atomically layered structure of MAX phase coupled with the metal and MAX phase layers, we engineer a hierarchical layered microstructure – where the metal and MAX phase layers are in direct competition with the internal interfaces within the MAX layers and atomically layered structure – taking advantage of the mechanisms of confined layer slip, Hall-Petch strengthening, and the competing deformation mechanisms to improve the strength of the system. By adjusting the combination of individual layer thicknesses, we can tune the mechanical properties of the metal-MAX phase multilayered nanolaminate system.

We begin by discussing the challenges associated with synthesizing MAX phase, followed by preliminary depositions on a Nb and Ti2AlC metal-MAX phase system which prefaced the overarching challenge of diffusion in the metal-MAX phase multilayered nanolaminates. To investigate and understand the diffusion, the next set of depositions focus on Ti and Ti2AlC metal-MAX phase multilayered nanolaminates to simplify the material system, as well as improve the plastic co-deformation between the metal and MAX phase systems. We utilize conventional direct current (DC) magnetron sputtering physical vapor deposition as well as a unique twin chamber deposition system, capable of automated atomic layer deposition and direct current magnetron sputtering physical vapor deposition without breaking chamber vacuum. We implement high temperature deposition, mixed low and high temperature deposition, as well as combined ALD-PVD depositions with amorphous Al2O3 layers to investigate the pathways to fabricate continuous metal-MAX phase multilayered nanolaminates. The multilayered depositions are characterized with XRD and TEM, and their mechanical properties are evaluated using nanoindentation and micropillar compression.

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