Tribology and Mechanical Behavior of Coatings and Engineered Surfaces
Room San Diego - Session E2-1-TuM

Mechanical Properties and Adhesion I

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

8:00am **E2-1-TuM-1 Indentation Behavior of Metal-Ceramic Multilayer Coatings: Modeling vs. Experiment***, Yu-Lin Shen,* University of New Mexico, USA **INVITED**

Thin films consisting of alternating metal and ceramic layers are an exciting subset of materials with many promising attributes. This presentation highlights our recent studies on mechanical characterization of such coatings using nanoindentation. We focus on aluminum (Al)/silicon carbide (SiC) nanolayers, which serves as a model system for investigating the constraining effect due to the highly mismatched mechanical properties of the constituents. How this structural heterogeneity responds to nanoindentation is a current a subject of active research. The development of complex deformation patterns underneath the indentation, dictated by the structural heterogeneity, can lead to various forms of local damage. Our studies focus on the employment of numerical finite element modeling to corroborate with experimental observations as well as to extract meaningful constitutive properties. Special attention is given to the analyses of (i) plastic deformation in the metal layers, (ii) cyclic indentation response and composite modulus measurement, (iii) indentation-induced delamination, and (iv) indentation-induced shear band formation.

8:40am **E2-1-TuM-3 Indentation Induced Delamination for Adhesion Measurements***, Megan J. Cordill, A Kleinbichler,* Erich Schmid Institute of Materials Science, Austria

Silicon nitride (Si_3N_4) is a frequently used passivation layer as well as an ion barrier in microelectronic devices. Its most outstanding properties are high fracture toughness and thermal stability, but in order to have a reliable device the adhesion to other thin films is of great importance. Qualitative tests like tape tests have shown that the adhesion of the brittle interface of Si_3N_4 and silicon glass (BPSG) is rather poor. In order to determine a quantitative measure of the adhesion of Si_3N_4 to BPSG other techniques such as nanoindentation are necessary. Indentation induces well-defined areas of delamination at the Si_3N_4-BPSG interface only when used in conjunction with a stressed overlayer of WTi. In this film system, the stressed overlayer helps the underlying Si_3N_4 film to buckle and supports the delaminated, brittle Si3N4 film from spalling from the substrate. Utilizing the delaminated areas and well established methods the adhesion of brittle thin films can be determined, which could not be measured before.

9:00am **E2-1-TuM-4 Intrinsic Stress in Polycrystalline Film: An Atomistic View***, Enrique Vasco,* Instituto de Ciencia de Materiales de Madrid, Spanish National Research Council (CSIC), Spain*; D Franco,* Departamento de Física de la Materia Condensada, Universidad Autónoma de Madrid, Spain*; E Michel, C Polop,* Departamento de Física de la Materia Condensada and Condensed Matter Physics Center (IFIMAC), Universidad Autónoma de Madrid, Spain

Most of the current applications based on films and coatings use continuous and compact polycrystalline films. They are easy to produce and integrate in many devices, cost-effective and suitable for common applications. However, their reliability suffers as the applications demand more resources, intensive use of medium properties or nonstandard working conditions. These limitations are due to the heterogeneous structure of the polycrystalline films, which are made up of misorientated grains separated by boundaries and other defects. Where the crystal lattice of the films has defects, a local field of intrinsic stress is generated. This stress field, which is reversible and cumulative with the working conditions, determines the short-range properties of the films and shortens the lifespan of devices.

The evolution of the intrinsic stress during the preparation of polycrystalline films has been investigated over decades. Nowadays, it is well known that the continuous compact films grow under compression, whose strength is closely linked with the density of grain boundaries (GB). However, the origin of the compression has not been clarified yet despite many models have been proposed. Recently, we measured the local distribution of residual intrinsic stress in polycrystalline films using a pioneering method[1]of nanoscale stress mapping based on AFM. Our results[2] demonstrated that, at odds with expectations, compression is not generated inside GBs, but at the edges of gaps where the boundaries intercept the surface. We report now a "definitive" model[2,3], wherein the compression is caused by Mullins-type surface diffusion towards the GBs, which generates a kinetic surface profile different from the equilibrium profile predicted by Laplace-Young. Where the curvatures of both profiles differ (mainly, at the edges of gaps where diffusing atoms accumulate), the intrinsic compression rises in the form of Laplace pressure. Our model[3] addresses successfully all the major evidences reported so far regarding the behavior of stress with experimental conditions. However, owing to the mesoscopic nature of our model, it is difficult to correlate it with atomistic processes that provide a physical vision (rather than mathematical one) of the phenomenon. In this work, we update our model in atomistic terms, namely, we address the origin and behavior of the intrinsic compression from the density of surface species (i.e., monomers and steps) and random walk diffusion with aggregation/nucleation leading to step-flow/second-nucleation growths.

1) C. Polop et al., Nanoscale **9**, 13938 (2017)

2) E. Vasco et al., PRL **119**, 256102 (2017)

3) E. Vasco et al., PRB 98, 195428 (2018)

9:20am **E2-1-TuM-5 Development of a Methodology for Measuring the Elastic Constants of Anisotropic Coatings Using Impulse Excitation Technique***, Elia Zgheib,* University of Technology of Troyes (UTT) and Lebanese University (UL), France*; M Slim, A Alhussein,* University of Technology of Troyes (UTT), France*; K Khalil,* Lebanese University (UL), Lebanon*; M Francois,* University of Technology of Troyes (UTT), France

New processes for obtaining advanced materials, in the form of coatings, are developing more and more to meet the socio-economic and the environmental needs. Coatings, as protective layers, improve the surface properties of a material. For example, in biomedical, elastic properties are must to be considered for the good insertion of the prostheses into the bone tissues. Thus, the deposit of Micro metric and/or Nano metric layers to obtain gradient functional materials is a promising approach for achieving impedance matching or reduction of stress concentrations on structures, such as prostheses or implants.

The project is part of the developments of Impulse Excitation Technique (IET) measuring constants of elasticity of thin layers [1, 2]. The IET is based on the analysis of vibrational frequencies created by an impact on a specimen. In this project, deposition will be developed using low-pressure process: PVD (Physical Vapor Deposition); and the parameters influencing the elasticity of the coatings will be identified. In the literature and for a designed anisotropic coating, no technique is available to go back to the values of the elastic constants of each layer and stack. This difficulty motivates the researcher to innovate methods and characterizations. What the project proposes is developing an inverse method using a multilayer model and the global analysis of vibration modes obtained by IET.

The methodology being used is a multi-scale approach to correlate the microstructural state of the coating and its resulting properties. The objectives that will be achieved rely on the development of IET measurement technique for mono and multi anisotropic coatings, by understanding the link between the parameters of elaboration, the physical-chemical properties, the microstructures and the elastic constants of the materials.

Keywords: Coatings, Elastic constants, Anisotropy, Multilayers, Impulse Excitation Technique, PVD.

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References:

[1] M.F. Slim, A. Alhussein, F. Sanchette, B. Guelorget, M. François, A new enhanced formulation to determine Young's and shear moduli of thin films by means of Impulse Excitation Technique, Thin solid films, 2017.

[2] M.F. Slim, A. Alhussein, A. Billard, F. Sanchette, M. François, On the determination of Young's modulus of thin films with Impulse Excitation Technique, Journal of Materials Research, 2016.

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