Advanced Characterization Techniques for Coatings and Thin Films
Room Royal Palm 1-3 - Session H1

Spatially-resolved Characterization of Thin Films and Engineered Surfaces

Moderators: Xavier Maeder, Empa, Swiss Federal Laboratories for Materials Science and Technology, Michael Tkadletz, Montanuniversität Leoben

9:00am **H1-4 Spatially Resolved Depth Profiling Of Residual Stress By Micro-Ring-Core Method***, **Marco Sebastiani,*** Roma TRE University, Italy

In this presentation, we will show some fundamental advances to the depth profiling capabilities for micro-scale ring-core focused ion beam (FIB) method. A new model, based on a single variable eigenstrain influence function approach, is developed and validated by comparison of FIB-DIC experimental results with synchrotron nano-diffraction experiments on the same reference sample (TiN PVD coating deposited by MS-PVD).

To this end, we revisit and revise the classical integral method proposed over quarter of a century ago. Instead of focusing on the residual stress that varies with every increment of hole-drilling, we concentrate on the reconstruction of eigenstrain, the invariant source of residual stress, and establish that the eigenstrain depth profile can be reconstructed by compact direct calculation, obviating the need for matrix formulation of the integral equation.

The method's sensitivity and robustness is further improved by the rational combination of multiple datasets obtained using different ring-core diameters. In this way, the approach has been shown to achieve depth resolution better than 50nm, opening up the prospect of reliable residual stress analysis at the nanometer scale.

9:20am **H1-5 Quantitative Depth Profiling from the First Nanometers Down to the Substrate within Minutes using RF GD-OES***, **Philippe Hunault,*** HORIBA Instruments, USA*; M Chausseau, K Savadkouei,* HORIBA Scientific, USA*; P Chapon, S Gaiaschi,* HORIBA Scientific, France

Glow Discharge Optical Emission Spectrometry (GD-OES) provides direct measurement of the chemical composition of materials as a function of depth and can be used to characterize various coatings, made of both thin and thick layers, conductive or non-conductive materials.

It consists in a pulsed radiofrequency glow discharge plasma source that is sputtering a large area of the material of interest and real time detection by a high resolution optical spectrometer of the sputtered species excited by the same plasma. All elements from H to U can be measured using this technique.

With its capability to perform depth profiling with a nanometric resolution and to go up to 150 µm deep into the sample within few minutes, GD-OES is an ideal tool to evaluate depth profiles on materials and to study interfaces between layers, diffusion processes or to optimize coatings processes. Many elements can be analyzed simultaneously, including Oxygen, Hydrogen, Deuterium, Carbon, Fluorine, Sulfur, Lithium… GD-OES is a versatile tool to study materials that complements other techniques such as XPS and SIMS.

Recent developments made possible using GD-OES for the direct determination of layer thickness and also for the analysis of odd shape samples.

Results obtained on various nm thin and thick coatings will be shown during this presentation: The use of RF GD-OES for the optimization of electroplating processes will be described with depth profiles of coatings on both inorganic and organic substrates and the direct determination of thickness using Differential Interferometry. How this technique can be used for Quality Control for the Aluminum packaging industry will also be shown with the help of real examples. Finally, we will discuss the latest results obtained for the characterization of the various coatings applied on drill bits which is a challenging sample by its shape and size.

9:40am **H1-6 Analysis of Thin Film Surface Stress Distribution using Raman Spectroscopy near Cohesive Cracks During Bending Tests***, **Newton Fukumasu,** G Francisco, R Souza,* University of São Paulo, Brazil

Stress distribution in a thin film is an important tool to control system performance in many applications. Usually, compressive stresses improve the wear behavior of the system, but the higher the compressive stresses, the higher the probability of coating adhesive failure. Stress calculation is based on the measurement of strain and present several pitfalls, frequently related to the elastic constants selected to correlate stains and stresses. This work aims contributing with the analysis of these constants in micro-Raman spectroscopy analyses. To this end, this work presents a technique to study the spatial distribution of the stresses at a coating surface near cracks generated by tensile stresses during bending tests. Literature reports that the stress state varies as a function of the distance from coating cohesive cracks, indicating that stresses are zero near the cracks. Local strains were measured based on the shift of the Raman spectrum. Measurements were conducted for a coated system composed of metal nitride films, deposited by sputtering, and ductile substrates. The stress evolution during the bending tests was reproduced numerically by means of simulations using the finite element method (FEM). Results were analyzed based on the correlation of the shift of the Raman spectrum and the stress evolution in bending tests, both during and after the test.

10:00am **H1-7 *In situ* Nanomechanical Characterization of Transition Metal Carbides***, M Chen,* ETH Zurich, Laboratory for Nanometallurgy, Switzerland*; D Sangiovanni,* Linköping University, IFM, Germany, Sweden*; J Wheeler,* ETH Zurich, Laboratory for Nanometallurgy, Switzerland*; **Suneel Kodambaka**, G Po,* University of California Los Angeles, USA **INVITED**

Refractory transition-metal carbides, owing to a mixture of ionic, covalent, and metallic bonding, exhibit high hardness, high stiffness, good resistance to wear, ablation, and corrosion, high-temperature mechanical strength along with good electrical conductivity. While these materials are exceptionally hard, their room-temperature structural applications have been limited by their brittleness. However, existing literature suggests that these carbides are not intrinsically brittle. Our recent nanomechanical tests carried out on sub-micrometer size carbide crystals have shown that these transition-metal carbides can undergo plastic deformation at room temperature.

In this talk, I will present results from our studies focused on understanding the mechanical deformation mechanisms operating in NaCl-structured TaC single-crystals. Using a combination of *in situ* scanning-electron-microscopy-based uniaxial micro-compression tests, *ab initio* molecular dynamics simulations conducted at temperatures up to 873 K, along with finite-element based modeling of discrete dislocation and crack dynamics, we determine the mechanisms leading to slip, dislocation nucleation and motion, and crack formation in TaC single-crystals. Our results provide new insights into the role of crystal orientation, size, and temperature on the correlation between plasticity and fracture in this class of materials.

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