Advanced Characterization Techniques for Coatings and Thin Films
Room Grand Hall - Session HP
Symposium H Poster Session

HP-1 Temperature Dependence of Nanocrystalline Aluminum Thin Film Elastic Constants by In-situ Brillouin Light Scattering and Picosecond Ultrasonics: Comparison to Molecular Dynamics*, **Philippe Djemia,*** LSPM-CNRS, France*; L Belliard,* INSP-UPMC, France*; H Zhang, Q Hu,* IMR-CAS, China*; F Challali, N Girodon-Boulandet, D Faurie,* LSPM-CNRS, France

We investigated the effective elastic constants in the temperature range [20°C, 600°C] of a polycrystalline aluminum thin film deposited on a silicon substrate by rf magnetron sputtering from Al target in Ar plasma discharge. From x-ray diffraction, Al polycrystalline film did not show strong crystallographic texture. The Brillouin light scattering (BLS) and the picosecond ultrasonics (PU) were complementary employed in combination with a furnace under high –vacuum (~10^{-5} mbar) or inert gas, to measure their acoustic and elastic properties. At room temperature, the Al film could be considered as nearly isotropic while increasing the temperature until 600 °C led to a continuous decrease of the elastic constants and an increase of the elastic anisotropy that was evaluated by a C_{11}/C_{33} ratio greater than one, of the in-plane (C_{11}) and out-of-plane (C_{33}) longitudinal effective elastic constants. Our results are found to be in a good agreement with previous experimental studies on bulk Al using standards ultrasounds equipment and our molecular dynamics theoretical estimates.

HP-2 High Resolution Full-field Curvature Measurement*, S Grachev,* ***Quentin Herault****, J Wang, I Gozhyk,* Saint-Gobain Recherche, France*; R Lazzari,* INSP-UPMC, France

A novel approach to curvature measurement will be presented. It allows for measuring curvatures below 10^{-7} m^{-1}. The method was successfully applied to observation of the first stages of growth of Ag by sputtering with unprecedented detail. The full-field capability allows for observation of complex forms resulting in variation of local curvature. The method was applied to a Mo film with inhomogeneous thickness distribution, which provided the curvature distribution maps. The poster will present the details of the method as well as its applications.

HP-4 In-situ High Temperature Characterization of DLC Films Using an Integrated Synchronized System*, M Rouhani,* National Chung Cheng University, Taiwan*; F Hong,* National Cheng Kung University, Taiwan*;* ***Yeau-Ren Jeng****,* National Chung Cheng University, Taiwan

An integrated synchronized system capable of mapping of depth-sensing indentation, spatially synchronized with a Raman spectrometer was applied to analyze microstructure, mechanical properties and surface roughness of DLC films over a specific area of the surface simultaneously. This integrated system was equipped with a high temperature chamber coupled with feedback control to make it possible to study the temperature effects on the mechanical properties and the microstructure evolution of the films, in ambient air up to 450̊C. A series of DLC films with different sp^3 content were deposited on Si substrates using filtered cathodic arc vacuum (FCVA) deposition system. Our study confirms the previous results that the thermal stability of the a-C films depends on their initial sp^3 content. The results show that the structural change is accompanied with profound increase in surface roughness of the films. The hardness of the films decreases with temperature increasing even before any changes in the microstructure of the films could be detected using Raman spectroscopy. The capability of the synchronized system enabled us to explore surface sensitive phenomenon of a-C film due to temperature rise that was not known before. Hence, our *in-situ* study showed for the first time that the surface sensitivity to temperature is greater than for the bulk of a-C films.

HP-6 Novel Methodology for the Evaluation of Mechanical Properties of Specific Crystalline Phases Present in Alumina Layers Formed by Plasma Electrolytic Oxidation (PEO) of Aluminium Alloys*, **Etienne Bousser,** A Yerokhin, A Gholinia, P Withers, A Matthews,* University of Manchester, UK

Aluminium alloys are widely used for their high specific strength but because of their lower hardness, these alloys often present a less than ideal resistance to surface wear. In order to improve the tribo-mechanical behaviour of these materials, electrolytic processes have been used for many years to promote the formation of protective oxide layers. More recently, Plasma Electrolytic Oxidation (PEO) has been shown to offer excellent wear performance through increased hardness due to the formation of hard crystalline phases during the oxide growth process at near-to-ambient bulk metal temperatures. However, the evaluation of the mechanical properties of the constituent phases in such oxide layers is difficult due to their complex microstructure. Indeed, these coatings are typically non-uniform with a shallow porous top layer which sits on a thicker, relatively dense layer comprising of a mixture of polycrystalline and amorphous oxide phases. In addition, complex multi-scale crack and pore networks are present over the entire thickness of the layer, making the evaluation of mechanical properties problematic using standard depth-sensing indentation methodologies. In this study, we present a novel approach for the precise evaluation of the mechanical properties of individual crystalline phases in the Al_2O_3 layers formed on aluminium alloys using Pulsed Bipolar PEO processes. The methodology presented in this poster is based on the statistical analysis of highly spatially-resolved mechanical property mapping of oxide layer cross-sections combined with high resolution characterization of the crystalline phases using Electron Back-Scattered Diffraction (EBSD). The EBSD was carried out on cross-sections prepared by Xe^+ ion Plasma FIB serial-sectioning and broad Ar^+ ion beam milling. In order to gain deeper insights into the processing effects on structure-property relationships, layers formed at different process times were evaluated.

HP-7 In situ High Temperature Fracture Toughness Evaluation of Hard Thin Ceramic Coatings by Means of a Micro-pillar Splitting Technique*,* ***Juri Wehrs,*** Platit AG, Switzerland*; J Best,* University of New South Wales, Australia*; M Polyakov, X Maeder,* EMPA - Swiss Federal Laboratories for Materials Science and Technology, Switzerland*; J Wheeler,* ETH Zürich, Switzerland*; M Morstein, B Torp,* Platit AG, Switzerland*; J Michler,* EMPA - Swiss Federal Laboratories for Materials Science and Technology, Switzerland

The fracture toughness at elevated temperatures is currently quite a difficult property to measure, but is a useful engineering variable for knowledge-based hard coating design. Most relevant applications of hard coatings, for example for metal cutting and forging tools, require usage at elevated temperatures. However several difficulties exist concerning toughness elucidation of small-scale specimens, mainly due to size effects, plasticity, residual stress effects and the influence of ion penetration from the sample fabrication process. Measuring at elevated temperatures magnifies the complexity of this task even further. Hence only few examples of high temperature fracture toughness measurements at high temperatures exist for small-scale samples.

In this study we explore the fracture toughness of a variety of arc-PVD coatings (CrN, AlTiN, AlCrTiN, and a AlCrN/SiNx nanocomposite) by means of a pillar splitting technique in the temperature range from RT to 500°C. The pillar splitting method for small-scale fracture toughness evaluation is inherently advantageous as the fracture event occurs under a sharp nanoindentation tip and in a region of material not significantly influenced from ion penetration and implantation. Therefore such a method is interesting for the study of brittle small-scale samples where ion implantation effects may be problematic. Further, unlike for other direct indentation techniques used to measure fracture toughness, the interface between coating and substrate does not interfere with the cracks that form, keeping the physics relatively simple.

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