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

HP-2 How Can the Icephobicity of an Engineered Surface be Screened by Means of Simple Laboratory Testing and Characterization?, *G de la Fuente, L Angurel,* CSIC-Universidad de Zaragoza, Spain*; C López-Santos, V Rico, A Borrás, A González-Elipe,* Instituto de Ciencia de Materiales de Sevilla (CSIC), Spain*; J Mora, P García,* ***Alina Agüero,*** Instituto Nacional de Técnica Aeroespacial (INTA), Spain

Ice formation and accretion present serious safety issues for aircraft, the automotive industry, signalling devices, railway systems, buildings, wind energy conversion plants, terrestrial power lines and many others. Considerable effort has been devoted to tackle this crucial yet challenging issue, mainly by developing anti-icing coatings that, based on the idea that a non-wetting material would diminish the water-surface contact and avoid the ice formation, link the hydrophobic properties of the materials with a supposed anti-icing function. However, it has been demonstrated that this rule is not always true, as in very humid environments ice can form on superhydrophobic materials.

The Canadian-European project“Super-IcePhobic Surfaces to Prevent Ice Formation on Aircraft” (PHOBIC2ICE) aims at producing durable icephobic surfaces by means of surface engineering and/or coatings. One of the main challenges faced by this project is to achieve screening laboratory tests capable of establishing if a given coating or engineered surface shows promising anti-icing properties, before having to run complex and expensive icing wind tunnel (IWT) or flying tests. Most published work in this domain use the measurement of the wetting angle and on the superhydrophobicity level of the surfaces as a pass or not pass criterion for further testing, despite that hydrophobicity and icephobicity are not necessarily correlated. The present work evaluates a simple test developed within the project, for measuring ice accretion, and compares it with a series of other simple laboratory tests and characterization techniques such as wetting angle, contact angle hysteresis, freezing delay, and roughness. Various surface treatments have been investigated in an attempt to correlate the results of these test results and the icephobicity of said materials. On the second stage of PHOBIC2ICE, the results will be compared with those obtained in an icing wind tunnel and flight tests.

HP-3 Pushing the Envelope in Variable Temperature Nanoindentation: High and Cryogenic Temperature Measurements, *N Randall, M Conte,* Anton Paar TriTec, Switzerland*; J Schwiedrzik, J Michler,* EMPA, Switzerland*;* ***Pierre Morel,*** Anton Paar, USA

One of the primary motivations for development of instrumented indentation was to measure the mechanical properties of thin films. Characterization of thin film mechanical properties as a function of temperature is of immense industrial and scientific interest. The major bottlenecks in variable temperature measurements have been thermal drift, signal stability (noise) and oxidation of/condensation on the surfaces. Thermal drift is a measurement artifact that arises due to thermal expansion/contraction of indenter tip and loading column. This gets superimposed on the mechanical behavior data precluding accurate extraction of mechanical properties of the sample at elevated/cryogenic temperatures. Vacuum is essential to prevent sample/tip oxidation at elevated temperatures and condensation at cryogenic temperatures.

In this poster, the design and development of a novel nanoindentation system that can perform reliable load-displacement measurements over a wide temperature ranges (from -150 to 800 °C) will be presented emphasizing the procedures and techniques for carrying out accurate nanomechanical measurements. This system is based on the Ultra Nanoindentation Tester (UNHT) that utilizes an active surface referencing technique comprising of two independent axes, one for surface referencing and another for indentation. The differential depth measurement technology results in negligible compliance of the system and very low thermal drift rates at high temperatures. The sample, indenter and reference tip are heated/cooled separately and the surface temperatures matched to obtain drift rates as low as 1nm/min at 800 °C without correction. Instrumentation development, system characterization, experimental protocol, operational refinements and thermal drift characteristics over the temperature range will be presented, together with a range of results on different materials.

HP-4 Surface and Sub-Surface Damage in Si and Ge Crystals after Nano-Machining, ***Jozef Keckes,*** Montanuniversität Leoben, Austria*; Z Zaprazny, D Korytar, M Jergel, Y Halahovets, P Siffalovic,* Slovak Academy of Sciences, Slovakia*; C Ferrari, C Frigeri,* CNR-IMEM Institute Parma, Italy*; I Matko, J Drga,* Slovak Academy of Sciences, Slovakia*; P Vagovič,* DESY, Center for Free-Electron Laser Science, Germany

Nano-machining methods like single point diamond turning (SPDT) and fly cutting (FC) are used to fabricate high-quality active surfaces of X-ray crystal optics. In this contribution, experimental results from Ge and Si surfaces will be used to demonstrate sub-surface damage (SSD) of the crystal lattices after various machining steps. The machined surfaces are characterized using atom probe microscopy, transmission electron microscopy (TEM), focused ion beam milling, scanning electron microscopy and Raman spectroscopy. The results reveal that the SSD and residual stresses correlate with the machining conditions. The morphology of surface ripples as well as a periodic variation of Raman peak shift are observed and correlated. TEM results are used to demonstrate the influence of the machining on the cross-sectional lattice damage.

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HP-6 Influence of Post-deposition Annealing on the Electrical Properties of Thin SiO2/a-Si:H/SiO2 Structures Obtained by Electron Cyclotron Resonance, ***David Mateos,*** Universidad Autónoma de Baja California, Mexico*; J Diniz,* University of Campinas, Brazil*; N Nedev, B Valdez, M Curiel,* Universidad Autónoma de Baja California, Mexico*; M Mederos,* Renato Archer Center for Information Technology, Brazil*; O Pérez, A Arias,* Universidad Autónoma de Baja California, Mexico

In this work results are presented for the deposition of thin multilayer structures, SiO2/a-Si:H/SiO2, by electron cyclotron resonance-chemical vapor deposition (ECR-CVD) technique. The ECR remote plasma systems present excellent characteristics that allow deposition of high quality uniform films at room temperature suitable for application in CMOS technology.

The depositions were carried out at an applied microwave power of 250 W under a gas pressure of 2.0 mTorr and a substrate temperature of 20°C. SiO2 film with thickness of ~6-8 nm were deposited using O2 and 2% SiH4 diluted in Ar as precursor gases. Hydrogenated amorphous silicon (a-Si:H) films with thickness of ~3-4 nm were deposited using the same flows of SiH_4 and Ar as in the case of SiO_2 but without O_2 flow. The films were deposited on p-type (100) c-Si substrates.

The film thicknesses in the multilayer structures were evaluated using the deposition rates of the a-Si:H and SiO_2. Individual layers of SiO_2 and a-Si:H were deposited on c-Si wafer and their thicknesses were determined ellipsometrically. As-deposited films were annealed at 800°C and 1100°C in N_2 atmosphere for 60 minutes.

Three-layer MOS structures were patterned by lithography and sintered in forming gas for 20 min. The influence of high temperature post-deposition annealing on the electrical properties of the structures was studied by current-voltage (I-V) and capacitance-voltage (C-V) measurements.

I-V measurements indicated that the high temperature annealing improves the gate dielectric properties. The C-V dependencies showed a correlation between the annealing temperature and the memory window of the structures. A possible explanation is that the high temperature annealing leads to structural modifications and formation of traps suitable to trap carriers. In addition, a strong improvement of the insulating properties of the SiO2 films was observed. Both changes may contribute to the observed memory effect. The obtained results show that the studied structures have a potential for application as gate insulators in non-volatile memory devices.

HP-7 Comparison of Three Methods for Ellipsometry Characterization of Thin Absorbing Films, ***Frank Urban,*** Florida International University, USA*; D Barton,* Retired, USA

Ellipsometry is a surface and film analytical technique which takes advantage of the fact that light reflecting from a surface undergoes a change in polarization state. The change in state results from the geometric structure and materials making up the reflecting surface. The predicted change in state can be determined through appropriate application of Maxwell's equations resulting in a mathematical model containing the surface descriptive parameters. In the common measurement scenario some of these parameters may be known and some, unknown, for example film thickness. Thus the method first requires fabrication of reflecting surfaces which result in tractable models. Next, measurements are made, at times spectroscopic, at multiple incidence angles, and on films of increasing thickness. Finally adjustments in the model and parameters are performed to put measurement and theory into agreement. It is well understood that these steps will depend upon the materials and thicknesses and other aspects of the reflecting surface. The purpose of the work to be presented is to make a direct comparison between different methods for determining the model and achieving agreement using the exact same films and measurements. The relative advantages and disadvantages of each method will be described. The three methods are that of Yamamoto which solves for pairs of measurements on growing films, the method in common use which seeks statistical agreement using the Levenberg-Marquardt algorithm to minimize mean square error, and the methods of the authors in the n-k plane which cast the problem into a deterministic mathematical expression which is solved numerically, typically by Newton-Raphson. Three different materials systems are explored; NiOx/Si, Cr/Si, and Ni/BK7. In each case the film is absorbing and ranges from a few nanometers up to approximately 30 nm in thickness. Differences in resolution, accuracy, model specificity, and difficulty of application will be presented.

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