Advanced Characterization, Modelling and Data Science for Coatings and Thin Films
Room Town & Country C - Session CM2-2-ThA

Advanced Mechanical Testing of Surfaces, Thin Films, Coatings and Small Volumes II

Moderators: Dr. Thomas Edwards, NIMS, Japan, **Matteo Ghidelli**, CNRS, France

1:20pm **CM2-2-ThA-1 Influence of Applied Deformation on Magnetic Properties of Ferromagnetic $Ni_{60}Fe_{40}$ Thin Films Deposited on Polymeric Substrate***, Alejandro Toledano Povedano **[alejandro.toledano.povedano@univ-poitiers.fr]**,* Institut Pprime - CNRS - ENSMA - Université de Poitiers, France*; Dominique Thiaudière,* Synchrotron SOLEIL, France*; Pierre Godard,* Institut Pprime - CNRS - ENSMA - Université de Poitiers, France*; Eloi Haltz,* Laboratoire des Sciences des Procédés et des Matériaux (LSPM) – CNRS, France*; Damien Faurie, Fatih Zighem,* Laboratoire des Sciences des Procédés et des Matériaux (LSPM) – CNRS, France*; Anny Michel, Pierre-Olivier Renault,* Institut Pprime - CNRS - ENSMA - Université de Poitiers, France

Metallic ferromagnetic thin films are key components in devices including sensors, data storage, and signal processing systems. With the rise of flexible electronics, understanding the relationship between magnetic properties and mechanical deformations in the low and high strain regimes is critical. These deformations induce homogeneous elastic strains as well as strain heterogeneities due to crystalline defects and cracks, impacting the magnetic properties of films through magneto-striction and dipolar interactions. This study focuses on how mechanical strain and controlled crack propagation affect the magnetic properties of thin films on polymer substrates. The research aims to reveal the relationship between controlled microstructural changes (residual stress, film thickness) and magnetic properties, from initial strain to crack onset and subsequent propagation. These insights are critical for developing flexible magnetic devices that maintain performance under mechanical stress. To investigate these effects, a multi-scale approach has been caried out thanks to a unique setup developed at Synchrotron SOLEIL (DiffAbs beamline). It combines four techniques to study, in situ, the crystalline and magnetic properties of the sample subjected to equibiaxial or sequenced uniaxial tensile testing: X-ray diffraction to monitor the local lattice strain, digital image correlation to measure macroscopic distortions, electrical resistivity to reveal the crack onset and Magneto-Optical Kerr Effect to track the evolution of magnetic reversal. Ni60Fe40 thin films with varying thicknesses (20 and 200nm) have been deposited by ion beam sputtering on flexible polymer substrates and characterised under strain with this setup. Deformation tests of Kapton/Mo/Ni60Fe40 systems highlight the important role of the magnetoelastic field, induced by the difference of the in-plane stress components, for multi-cracking dynamics. The study also examined different film thicknesses to determine whether these variations were linked to fragmentation effects or magnetoplasticity. These findings show how crack density (which varies with thickness) influences the material's magneto-mechanical properties.The hysteresis loops initially show a square shape. As applied deformation increases, the loops change and exhibit features typical of a direction that resists magnetization, attributed to the negative magnetostrictive coefficient of Ni60Fe40. Beyond the maximum of the lattice strain, the loops appear to return to a square shape.

1:40pm **CM2-2-ThA-2 The Local Electrical Fingerprint of Deformation and Growth -Induced Defects in Alloys***, Hanna Bishara **[hbishara@tauex.tau.ac.il]**,* Tel Aviv University, Israel **INVITED**

A microstructural defect, whether spontaneously or intentionally induced, impacts the electrical properties of its surroundings. Defects dominate the electrical behavior of materials only when they become sufficiently dense. Therefore, capturing the defect's electrical characteristics is usually performed on a macroscopic scale, leading to averaging over multiple defect's types. This prevents studying the structure-properties relations in defects. This talk provides advanced electrical characterization methods of individual defects on surface and within the volume of bulk and thin film alloys.

The presentation initially introduces an experimental procedure to measure the local electrical resistivity of defect segments - with high sensitivity and spatial resolution *in-situ* scanning electron microscopy (SEM). The studied defects, i.e. pure and segregated grain boundaries (GBs), dislocations, stacking faults, and phase boundaries are either growth-controlled or deformation-induced. The segments are chemically and structurally characterized by electron backscatter diffraction (EBSD), transmission electron microscopy (TEM), energy dispersive spectroscopy (EDS), and atom probe tomography (APT), in addition to molecular dynamics (MD) simulations.

In the context of grain boundaries (GBs), we report that the GB resistivity spans over a spectrum of values, depending on the boundary's excess volume. The resistivity values might increase by an order of magnitude due to segregation effects in metallic systems. However, segregation-influenced complexions are found to boost the electrical conductivity of semi-metallic materials. Additionally, the talk relates to the formation and electrical characterization of near-surface dislocations in brittle Heusler alloys.

Revealing the contribution of different GB types to electrical resistivity would pave the path for predicting the electrical degradation of materials upon controlled mechanical deformation. In addition, it allows a novel defect engineering to optimize the performance of conductors and functional alloys.

2:20pm **CM2-2-ThA-4 On the Effect of Thin Film Residual Stress on the Crack Propagation Resistance of ALD Coated Nano-Ceramics***, Edoardo Rossi,* Università degli studi Roma tre, Dipartimento di ingegneria Civile, Informatica e delle Tecnologie Aeronautiche., Italy*; **Marco Sebastiani [seba@uniroma3.it]**,* Università degli studi Roma Tre, Dipartimento di Ingegneria Civile, Informatica e delle Tecnologie Aeronautiche, Italy

The present work aims at investigating the effects of Atomic Layer Deposition (ALD) coatings on 3D printed ceramic micro-pillars, which were produced by Two-photon polymerization-direct Laser Writing (TPP-DLW). With a uniform 50 nm layer of Al_2O_3 under varying processing conditions (Plasma Enhanced-ALD at 200 °C, Thermal ALD at 200 °C, and 350 °C), the study first evaluated how these coatings influenced the retainment of fracture toughness through the splitting of glassy carbon (GC) pillars across a spectrum of Relative Humidity levels (below 5% and above 60%). Then, incorporating spatially resolved stress measurements through Focused Ion Beam (FIB) ring-core analysis, the specific interface effects of the coatings on the crack propagation process were investigated. A corresponding investigation of lithographically produced fused quartz micro-pillars treated with the same ALD parameters provided a comparative foundation to gauge the coatings' effectiveness in enhancing the composite fracture toughness.

Additionally, the research detailed how the found residual stresses within the ALD coatings, significantly varying depending on the deposition temperature, are critical for the understanding of crack initiation and propagation mechanisms, suggesting that the observed reduction in fracture toughness, when compared to undefective, uncoated pillars under similar humid conditions, might be attributed to premature crack tip opening.

The research clarified these interplayed dynamics with the coating's stresses through the silica system's response to ALD coatings, significantly improving the baseline crack resistance. Indeed, uncoated silica experience an approximate 134% increase in fracture toughness for a 50 nm deposition at 200 °C, while a 100 nm coating at 300 °C resulted in around a 165% enhancement. This illustrates how interface engineering (deposition temperature and induced stresses from ALD coatings) can fine-tune fracture toughness in 3D TPP micro-ceramics, depending highly on the substrate material and surface defects (likely missing in lithography silica structures).

2:40pm **CM2-2-ThA-5 Micromechanical Testing of Ceramic Coatings for Nuclear Applications up to 1000°C***, **Dong (Lilly) Liu [dong.liu@eng.ox.ac.uk]**,* University of Oxford, UK **INVITED**

Multi-layered ceramic coatings, such as SiC and PyC, have been used to encapsulate spherical nuclear fuel kernels for use in the next generation of nuclear fission reactors. These coatings are typically between 30 µm to 100 µm thick and will subject to harsh environments such as elevated operation temperatures and neutron radiation during service. It is important to acquire local mechanical properties of these individual coating layers as well as the interfacial strength between the coatings for better understanding of their structural integrity and to support performance modelling. In this work, nanoindentation tests were carried out on SiC and PyC coatings over a range of temperatures from ambient to 1000°C with and without in-situ SEM imaging. The change of modulus and hardness as a function of temperature will be presented and the challenges associated with the high-temperature tests will be discussed. In addition, micro-cantilever bending method was utilized to evaluate the interfacial strength between the SiC and PyC coatings. During the coating deposition process (chemical vapour deposition), residual stresses were generated in the coatings and affected the local properties. Therefore, the residual stresses in each coating layer were characterised by focussed-ion-beam digital image correlation (FIB-DIC) method on unirradiated and neutron irradiated coatings where the magnitude of residual stresses are further modified due to radiation induced dimensional changes. The local mechanical properties and residual stresses measured are correlated with the coating deposition process, radiation damage and 3D microstructure generated using FIB tomography based on conventional Ga+ FIB and Plasms FIB.

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