Topical Symposia
Room Pacific C - Session TS2-1-TuA

Thin Films on Polymer Substrates: Flexible Electronics and Beyond

Moderators: Oleksandr Glushko, Erich Schmid Institute of Materials Science, Austria, **Barbara Putz**, Montanuniversität Leoben, Leoben, Austria

1:40pm **TS2-1-TuA-1 in Situ Observation of Strain Transfer and Crack Formation in Evaporated and Printed Thin Films and Devices on Compliant Substrates***, **Patric A. Gruber (patric.gruber@kit.edu)**, N. Misra, T. Haas , S. Yi, B. Kim,* Karlsruhe Institute of Technology (KIT), Germany **INVITED**

Compliant substrates enable the fabrication of flexible electronics for numerous applications like flexible displays, solar cells, batteries or wearable/biocompatible electronics. However, the reliability of such devices is limited by the stretchability of the inorganic components. So far, little experimental work has been carried out to investigate the mechanical properties of thin inorganic films on compliant substrates at high strains and cyclic loading. Here, we present experimental results for the flow stress, fracture strain and fatigue behavior of evaporated and printed Ag films as well as printed thin film transistors on compliant substrates. The film systems have been tested by a synchrotron-based tensile testing technique (up to 10% total strain) as well as cycling loading (50 Hz, strain amplitude up to 2.5%) and have been characterized by SEM and FIB microscopy. The synchrotron experiments yield the stress evolution and strain transfer within the film systems whereas the cyclic tests give the fatigue lifetime. On the other hand, *in situ* electro-mechanical testing, *in situ* tensile tests in the SEM and stationary FIB investigations reveal the evolution of electrical performance, crack morphology and crack density as well as fatigue damage in the individual films. First, results of electro-mechanical testing of printed and evaporated Ag films will be presented. Electrical conductivity and mechanical reliability are investigated with respect to the inherently nanoporous microstructure, and are compared to those of evaporated Ag films of the same thickness. It is shown that there is an optimized nanoporous microstructure for inkjet-printed Ag films, which provides a high conductivity and improved reliability. It is argued that the nanoporous microstructure ensures connectivity within the particle network and at the same time reduces plastic deformation and the formation of fatigue damage. Furthermore, results on printed In_2O_3 thin film transistors are presented. Here, the interplay of the polymer substrate and solid polymer electrolyte with the metallic and ceramic interlayers within the transistor structure will be discussed based on the strain evolution within the individual layers determined from the in situ synchrotron experiments.

2:20pm **TS2-1-TuA-3 Electrical Resistance During Cyclic Loading of Conductive Coatings – What Information is Hidden in the Data?***, **David Gebhart (david.gebhart@oeaw.ac.at)**, M. Cordill,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben, Austria

An increase in electrical resistance during cyclic loading of metallic films on polymer substrates is mainly a function of crack density in the film, mean crack length (and its distribution), and mean crack depth. Those factors cannot be separated in a single data point but an ensemble of datapoints may show trends that can be attributed to single factors. With a material system of a 150 nm Au coating on polyimide (Upilex-S; 50 µm), with and without a 30 nm Cr interlayer, a thorough analysis was done on information contained within electrical data obtained during cyclic tensile straining. With strain-controlled sinusoidal loading, twenty 4-wire resistance data points were recorded per cycle. By analyzing peak shape evolution, i.e. full width at half maximum values, cycle amplitudes, and curves fitted to minima and maxima points of each cycle, various damage initiation and propagation events could be identified and a distinction could be made between through-thickness cracking and surface necking. The findings are validated with imaging and characterization methods. Easy to implement and easily automated methods are presented, which can be used retroactively on acquired data, to make an argument for the effectiveness of deep analysis of electrical resistance data in fatigue investigations of conductive coatings.

2:40pm **TS2-1-TuA-4 Plasma Surface Activation of Epoxy Painted Polymer Composites to Enhance Adhesion of PVD Coatings***, **Nicolas Ranger (nicolas.ranger@oerlikon.com)**,* Oerlikon Balzers/IRCER, France*; C. Jaoul, P. Tristant,* IRCER, France*; T. Maerten,* Oerlikon Balzers, France*; S. Belveze,* Oerlikon Balzers , France*; S. Guimond,* Oerlikon Balzers, Liechtenstein*; M. Cavarroc,* Safran Tech, France

Polymer-based composites materials are increasingly employed in aircraft industry as replacement of metallic components due to their excellent specific properties (strength- and stiffness-to-density ratios). But aircraft surfaces are subject to harsh environmental conditions (rain, sand, gas, etc.) detrimental to polymer composites lifetime. As a result, deposition of hard ceramic films is required to protect them. PVD coating are envisioned but adhesion of such films is known to be low on these substrates [1].

Surface pre-treatments are therefore critical to improve the adhesion of coatings on polymer composite. Nowadays, it is commonly accepted that the enhancement of the adhesion of a coating is related to the density of nucleation sites at the polymer surface [2-3]. One simple way to quantify the increase of nucleation sites is by measuring surface energy.

The aim of the present study is to evaluate the effect of low pressure Ar, N_2 and O_2 plasmas on epoxy painted carbon fiber reinforced composite surface as adhesion-promoting treatment. Various plasma conditions are tested by changing parameters like gas mixture, bias voltage and duration. The surface energy is measured by contact angle measurements. These measurements are complemented by chemical analysis (XPS) and surface morphology observations (Profilometer, SEM, confocal microscopy) to understand the surface modification mechanisms.

Finally, the most promising plasma conditions as adhesion-promoting treatment are applied prior to the deposition of a titanium film deposited by magnetron sputtering. The adhesion is, then, evaluated using conventional methods (scratch and pull-off tests) to quantify the benefit of the pre-treatment.

Bibliography

[1] F. Awaja, M. Gilbert, G. Kelly, B. Fox and P. Pigram, "Adhesion of polymers," *Progress in Polymer Science,* vol. 34, no. 9, pp. 948-968, 2009.

[2] F. Faupel, T. Strunskus, M. Kiene, A. Thran, C. v. Bechtolsheim and V. Zaporojtchenko, "Fundamental apsects of polymer metallization," *Materials Research Society Symposium - Proceedings,* vol. 511, pp. 15-26, 1998.

[3] E. M. Liston, L. Martinu and M. R. Wertheimer, "Plasma surface modification of polymers for improved adhesion: A critical review," *Journal of Adhesion Science and Technology,* vol. 7, pp. 1091-1127, 1993.

3:00pm **TS2-1-TuA-5 MOKE-XRD Experiment for the Study of Magnetomechanical Properties of Thin Films Deposited on Stretchable Substrates***, H. Mahmoud,* Université Sorbonne Paris, Université de Poitiers—CNRS, France*; **Damien Faurie (faurie@univ-paris13.fr)**,* Université Sorbonne Paris, France*; P. Godard,* Université de Poitiers—CNRS, France*; D. Thiaudière,* Soleil Synchrotron, France*; P. Renault,* Université de Poitiers—CNRS, France*; F. Zighem,* Université Sorbonne Paris, France

Stretchable/flexible electronics has been a rapidly growing field for several years. In particular, magnetic systems realized on stretchable/flexible substrates are of increasing interest for their potential applications [1-2]. These will be subjected to large strains during their use. It is therefore important to understand the different phenomena involved at very large strains in order to estimate the durability of their functional properties, through the study of elementary magnetomechanical phenomena [3].

In this context, we have developed an original experiment coupling *in situ* Magneto-Optical Kerr Effect (MOKE) magnetometry, biaxial traction, digital image correlation on the DiffAbs beamline of the SOLEIL synchrotron, in order to follow the evolution of the magnetic quantities at different mechanical regimes (elastic domain, plasticity, multicracking) (figure 1 (a), supplementary material). This device has been used to study model thin films of Cobalt (20 to 100 nm) deposited on Kapton (polymide) substrate. The evolution of the magnetization curves as a function of the applied strain was analyzed in order to estimate the evolution of the coercive field, the remanent magnetization and the saturation field. This analysis has been correlated to the different deformation micromechanisms estimated thanks to the monitoring of the elastic strains by X-ray diffraction (figure 1 (b), supplementary material).

Through this presentation, we will show the instrumental development of the device (biaxial tensile machine coupled to the magnetometer) as well as the mechanisms underlying the measured properties according to the type of loading (equibiaxial or complex). The perspectives of these first studies will also be discussed (understanding of the physical mechanisms, effects of lateral nanostructuring, ...).

[1] D. Makarov, M. Melzer, D. Karnaushenko, O.G. Schmidt, Applied Physics Reviews 3 (1), 011101 (2016)

[2] F. Zighem, D. Faurie, Journal of Physics: Condensed Matter 33 (23), 233002 (2021)

[3] S. Merabtine, F. Zighem, D. Faurie, A. Garcia-Sanchez, P. Lupo, A. Adeyeye, NanoLetters 18, 3199 (2018)

4:00pm **TS2-1-TuA-8 Nanoscale Deformation Mechanisms in Thin Film Metallic Glasses Explored by in-Situ SEM With Digital Image Correlation***,* ***Oleksandr Glushko (oleksandr.glushko@unileoben.ac.at)****, C. Mitterer, J. Eckert,* Montanuniversität Leoben, Austria

Digital image correlation (DIC) is a powerful technique allowing detailed mapping of local strain distributions from the images of deformed surface. Spatial resolution of DIC is restricted only by the pixel size of the image and quality of random speckle pattern on the surface. Here we demonstrate the capabilities of DIC to capture propagation of shear bands in thin film metallic glasses with spatial resolutions down to few tens of nanometers.

PdSi metallic glass films were sputter deposited on polyimide substrate and then covered with randomly distributed nano-sized Indium islands. Additionally, the films were pre-structured with specific FIB-milled patterns in a way that the formation of cracks is locally prohibited but in-plane shear bands can freely propagate. Straining experiments were performed in-situ in SEM and GOM Correlate software was employed for DIC analysis. Shear bands propagating within the film plane do not necessarily lead to appearence of surface traces and thus cannot be detected on the SEM images with a naked eye. With DIC analysis shear bands are clearly visualized as bands of extremely localized strains, significantly exceeding the strains in the rest of the film. Local strains before and after generation of shear bands were carefully measured and an analogue of von Mises yielding criterion is formulated. Additionally, provided analysis proved that shear bands are “cold” during operation, i. e the temperature on the surface stays far below the glass transition temperature. Demonstrated technique of combining pre-patterned polymer-supported films with in-situ SEM straining and DIC is shown to be extremely effective to capture microplasticity phenomena with nanoscale resolution.

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