Advanced Characterization Techniques for Coatings and Thin Films
Room Royal Palm 4-6 - Session H2-2
Advanced Mechanical Testing of Surfaces and Coatings

Moderators: Benoit Merle, Friedrich-Alexander-University Erlangen-Nürnberg (FAU), Marco Sebastiani, University of Rome "Roma Tre"

1:30pm **H2-2-1 Mechanical Properties of High-strength Low-weight Truss Structures Fabricated by 3D Direct Laser Writing**, ***Ruth Schwaiger,*** Karlsruhe Institute of Technology (KIT), Institute for Applied Materials (IAM-WBM), Germany **INVITED**

Cellular materials with designed architectures are fabricated with the goal to design new materials with high strength and low density. In our approach, polymeric truss structures with characteristic features in the micro-to-nanometer range are fabricated by 3D direct laser writing and then coated using thin film deposition techniques. Annealing treatments as well as deposition of alumina or metal coatings are shown to enhance the strength of the structures.

Nanomechanical testing methods are used to investigate the deformation behavior of the fabricated structures. As demonstrated by push-to-pull tensile tests, annealing can increase the material strength by up to a factor of 10. However, it is found that this increase of strength cannot be fully transferred to architected materials, such as tetrahedral truss structures, because of stress concentrations at the truss nodes. Failure of the truss structures due to buckling and subsequent fracture at the truss nodes as well as a pronounced recovery upon unloading was observed. Cyclic tests showed energy dissipation, which is a function of progressively failing ligaments. Deformation and failure as well as the strategies to improve the properties of 3D microarchitectures will be discussed.

2:10pm **H2-2-3 An improved Nanoindentation Method to Measure Residual Stress and Elastic Moduli of Freestanding Multilayer Thin Films**, ***Marco Sebastiani,*** *M Ghidelli,* Roma TRE University, Italy

In this work, we developed an improved nano-mechanical characterisation procedure of freestanding bilayer (Au-TiW) micro-cantilevers and double clamped beams, for applications as Radio Frequency (RF)-switches Micro-Electromechanical Systems (MEMS). The micro-beams are deflected by using nanoindentation in dynamic stiffness measurement mode, in order to extract the elastic modulus and the residual stresses of both layers. Firstly, the classic beam theory has been implemented for bilayer cantilevers enabling the extraction of elastic moduli. Then, residual stresses are estimated by deflecting double clamped beams, while implementing new analytical models for a bilayer system. The obtained elastic moduli are consistent with the average ones obtained for a single layer micro-cantilever and with nanoindentation results for TiW and Au homogeneous films. The residual stresses are in agreement with the values obtained from the double slot Focused Ion Beam (FIB) and Digital Image Correlation (DIC) procedure, providing an alternative and portable way for the assessment of residual stresses on composite double clamped micro-beams.

2:30pm **H2-2-4 *In Situ* FIB-SEM DIC and Synchrotron XRD Analysis of the Mechanical Degradation of a Uniaxially Loaded Copper-Tungsten Nano-Multilayer**, ***León Romano Brandt***, *E Salvati, C Papadaki, H Zhang, S Ying, T Sui, A Korsunsky,* University of Oxford, UK

Thin coatings and multi-layers with individual layer thickness down to a few nanometres enable the design of materials with novel and carefully tailored thermal, mechanical, and functional properties. These properties arise as a consequence of the confinement of inhomogeneous mechanical, chemical and electromagnetic fields within nano-scale strata and nano-grains of material. External mechanical loading can cause structural damage and significantly decrease or undermine the performance of coating systems. It is therefore important to develop methods for quantifying material specific parameters, as well as attain a fundamental understanding of the mechanisms determining the mechanical response of thin films at the micro- and nanoscale and transform this fundamental understanding into generally applicable models.

The analysed copper-tungsten nano-multilayers with individual layer thicknesses of 18/6 nm and a total thickness of 240 nanometres are particularly attractive for applications as heatsinks in microelectronics. The micro- and nano-mechanical analysis of this material requires a variety of complex methods and techniques capable of precisely manipulating and resolving samples at these scales. *In situ* scanning electron microscopy (SEM) image sequences recorded during deformation of samples prepared by focussed ion beam (FIB) milling were interpreted using digital image correlation (DIC) to determine displacement and total strain fields. On this basis, a viable method is proposed for determining fracture toughness of thin nano-multilayers and other key properties of sub-micron thin films.

A very powerful tool for the determination of the elastic strain component of multilayers is synchrotron X-ray diffraction (XRD). We used this technique for *in situ* strain mapping regions of interest while applying stretching to the coating substrate. Based on the obtained strain maps, a description is proposed of the stress-strain state in hard brittle coatings on soft polymer substrates under substrate stretching.

2:50pm **H2-2-5 Synchrotron Nano-diffraction Studies of Ex-situ and In-situ Indented Thin Films: Microstructure and Stress Analysis**, ***Juraj Todt,*** Montanuniversität Leoben, Austria; *C Krywka,* Helmholtz-Zentrum Geesthacht, Germany; *M Burghammer,* European Synchrotron Ratiation Facility, France; *J Keckes,* Montanuniversität Leoben, Austria

Mechanical testing of small features such as thin films and surface regions is a challenging task, especially in the case of microstructurally complex systems exhibiting property gradients at the sub-micrometer scale. Since indentation is a popular technique, there has been considerable effort to model the rather complex underlying mechanical interactions. However, it has been difficult to find experimental validation for these numerical studies, due to the lack of adequate experimental methods.

In recent years cross-sectional scanning X-ray nano-diffraction at synchrotrons has been established as a powerful technique for structural analysis of thin films. Its key feature is a high spatial resolution, usually below 100 nm, offering insight into depth-gradients of phase composition, preferred orientation, morphology and stresses that evolve as a consequence of self-governed film growth or due to deliberate process control.

In the presented work, its capability to assess these sub-micrometer gradients is exploited as a means to study the microstructural changes and stresses induced during the indentation of thin films. This covers the extent, shape and magnitude of multi-axial stress fields during or after their formation, localized reorientation of grains, generation of defects, as well as their relation to the resulting failure modes. Multiple cases of multi-layered and monolithic metallic and ceramic thin films that were indented either ex-situ or in-situ will be discussed. Some of the presented cases will also be compared to respective FE analyses, highlighting areas for their improvement.

[1] Keckes J, Bartosik M, Daniel R, Mitterer C, Maier G, Ecker W, et al. (2012), X-ray nanodiffraction reveals strain and microstructure evolution in nanocrystalline thin films, *Scr Mater* **67**, 748–51.

[2] Stefenelli M, Daniel R, Ecker W, Kiener D, Todt J, Zeilinger A, et al. (2015) X-ray nanodiffraction reveals stress distribution across an indented multilayered CrN/Cr thin film *Acta Mater* **85**, 24-31.

[3] Zeilinger A, Todt J, Krywka C, Müller M, Ecker W, Sartory B, Meindlhumer M, et al. (2016) In-situ Observation of Cross- Sectional Microstructural Changes and Stress Distributions in Fracturing TiN Thin Film during Nanoindentation, *Sci Rep* **6**, 22670.

3:10pm **H2-2-6 Cross-sectional Microstructure and Mechanical Behaviour of As-deposited and Oxidised CVD TiB_2 Hard Coatings Determined by X-ray Nanodiffraction and Micro-mechanical Tests**, ***David Gruber,*** *M Tkadletz, N Schalk,* Montanuniversität Leoben, Austria; *B Sartory,* Materials Center Leoben Forschung GmbH (MCL), Austria; *C Mitterer, J Keckes,* Montanuniversität Leoben, Austria

In multilayered hard protective coatings used for industrial cutting operations, the functional behaviour depends on the properties of individual sublayers and interfaces.

In this contribution, mechanical properties, residual stresses and the cross-sectional microstructure of TiN/TiB_2 protective coatings on WC-Co substrates are analysed in as-deposited state and oxidised at 700°C in ambient air.

Synchrotron X-ray nanodiffraction is used to investigate gradients of phases and residual stresses as a function of coating depth in order to assess oxidation-induced changes in stress state and observe oxide formation.

In addition, in-situ micromechanical tests on micro-cantilevers, machined by focused ion beam milling of the TiB_2 and TiN sublayers are performed in the scanning electron microscope in order to determine Young's moduli and fracture stresses.

The experimental results indicate a complex nature of oxidation-induced degradation at the coating cross-section and allow to quantitatively evaluate changes in important coating parameters.

3:30pm **H2-2-7 Fundamental Mechanical Properties of Simple- and Pt/Ir-modified-Aluminide Diffusion Coatings after Thermocyclic Exposure**, ***Ceyhun Oskay***, *M Galetz,* DECHEMA-Forschungsinstitut, Germany; *H Murakami,* National Institute for Materials Science, Japan

The oxidation protection of Ni-based superalloys under service conditions relies on the mechanical and chemical stability of aluminide diffusion coatings, which provide the necessary Al-reservoir for the protective oxide scale formation. During exposure at high temperatures, the existing chemical gradient between the coating and substrate causes an alteration of the microstructure. Microstructural degradation of such coatings not only affects the chemical lifetime, but also influences the mechanical durability of the system. Therefore determination of the mechanical properties of simple- or modified-aluminide coatings is a significant step for precise lifetime assessment. The change in the microstructure is especially important for thin-walled components, for which the mechanical contribution of the coating gains importance.

In this study, simple aluminide coatings were deposited on four-point-bending flexural test samples of a 2nd generation Ni-based superalloy via a CVD-process. Pt- and Pt/Ir-modified aluminide coatings were manufactured on PWA 1483 samples via a two-step process involving electrodeposition and subsequent pack-aluminizing. Afterwards the samples were exposed to thermocyclic oxidation (1h cycles) at different maximum temperatures (highest 1100°C) for various durations (max. 1000 cycles). Fracture strain, hardness and elastic modulus were determined after the exposure at room temperature (RT) via four-point-bending flexural test with in-situ acoustic emission measurement and nanoindentation respectively. Consequently, the fundamental mechanical properties were correlated with the corresponding Al-concentration.

Al-depletion during thermocyclic exposure causes an increase in the fracture strain. Further depletion triggers the formation of the more ductile γ'-phase, through which the coating becomes able to deform plastically with the substrate at RT, rather than showing premature brittle fracture. Elastic modulus and hardness decrease within the single-phase region. However within the two-phase stability region, the formation of the γ'-phase causes an increase of both properties.

3:50pm **H2-2-8 Fast Nano-mechanical Property Mapping using XPM on Nano-crystalline Structures**, ***Anqi Qiu***, *D Vodnick,* Hysitron, Inc., USA

Surface properties of nano-crystalline materials have been studied intensively using various spectroscopy and microscopy techniques. But due to the sizes of the crystalline structures, it was difficult to map the deformation behaviors of nano-crystalline materials due to the continuum behavior of stress. With the development of ultra low noise XPM technique, nano-mechanical behaviors of nano-crystalline materials show good correlation between the mechanical behavior map and the crystallographic characterization. This gives a new direction of studying nano-crystalline plasticity and grain boundary mediated deformation.

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