Tuesday Morning, May 21, 2024

Advanced Characterization, Modelling and Data Science for Coatings and Thin Films
Room Palm 3-4 - Session CM2-2-TuM

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

Moderator: Matteo Ghidelli, CNRS, France

8:40am **CM2-2-TuM-3 Approaches for Circumventing FIB Artefacts in Small Scale Fracture Testing**, ***Eloho Okotete (eloho.okotete@kit.edu)***, *S. Lee, S. Brinckmann, C. Kirchlechner,* Karlsruhe Institute of Technology, Germany

Miniaturization has resulted in the production of small components ranging from a few nanometres to a few hundred micrometres, which are used in both consumer and industrial applications. To measure the fracture properties of thin films and interfaces at these length scales, geometries fabricated by Focused Ion Beam (FIB) are currently used. However, this method of milling can introduce impurities at the notch, which may affect the reliability of fracture measurements. To minimize the role of artefacts in small scale fracture studies, stable crack growth geometries are used, through which a sharp crack can be grown from a FIB notch. Another way to reduce the effects of FIB damage on test samples is by using inert notching ions.

In this talk, various methods to reduce FIB artefacts' role are presented systematically. In the first part, we discuss the study of fracture in single crystalline silicon notched with an inert ion source using a bridge notched single cantilever beam geometry that has recently been reported to demonstrate crack arrest. Furthermore, we use a new stable crack growth geometry to measure the fracture toughness of the hard-coating substrate interface [2]. The results indicate that cantilevers with deep notches and thin material bridges exhibit crack arrest, and the fracture toughness of cantilevers falls within the expected range for single crystalline silicon. In the new geometry, it is seen that the crack driving force decreases with crack extension in the geometry, preventing catastrophic failure. As a result, a natural crack is formed from the FIB notch, and final fracture occurs after the crack has grown beyond the region of the FIB milled notch. This reduces the influence of FIB-induced artefacts such as residual stresses due to ion implantation, finite notch radius, crystalline defects, and redeposition on the fracture toughness of materials tested at small length scales.

[1] Y. Zhang, M. Bartosik, S. Brinckmann, S. Lee, C. Kirchlechner, Direct observation of crack arrest after bridge notch failure: A strategy to increase statistics and reduce FIB-artifacts in micro-cantilever testing, Materials & Design 233 (2023) 112188.

[2] E. Okotete, S. Brinckmann, S. Lee, C. Kirchlechner, How to avoid FIB-milling artefacts in micro fracture? A new geometry for interface fracture, Materials & Design 233 (2023) 112134.

9:00am **CM2-2-TuM-4 Influence of Annealing-Induced Substrate Element Diffusion on the Microstructure and Mechanical Properties of TiN/TiCN Coatings Synthesized using Chemical Vapor Deposition**, ***Fabian Konstantiniuk (fabian.konstantiniuk@unileoben.ac.at)***, *M. Schiester,* Christian Doppler Laboratory for Advanced Coated Cutting Tools at the Department of Materials Science, Montanuniversität Leoben, Austria*; M. Tkadletz,* Department of Materials Science, Montanuniversität Leoben, Austria*; C. Czettl,* CERATIZIT Austria GmbH, Austria*; N. Schalk,* Christian Doppler Laboratory for Advanced Coated Cutting Tools at the Department of Materials Science, Montanuniversität Leoben, Austria

TiN/TiCN deposited by chemical vapor deposition (CVD) is widely used as hard coating system for cemented carbide cutting tools, typically under an Al_2O_3 top layer. During the deposition of the Al_2O_3 top layer, the underlying TiN and TiCN layers are exposed to high temperatures. Therefore, the present study focuses on the influence of this Al_2O_3 deposition step, which is mimicked by a vacuum-annealing treatment, on the microstructure and mechanical properties of the TiN/TiCN coating. By applying advanced characterization techniques such as scanning electron microscopy (SEM), electron backscatter diffraction (EBSD), atom probe tomography (APT), and micro-mechanical bending tests on both, as-deposited and annealed coatings, changes in the microstructure and mechanical properties were studied. It was found that W and Co diffusion takes place along the TiN and TiCN grain boundaries from the substrate into the coating. While the hardness, Young's modulus, and fracture toughness remained unaffected by the annealing treatment, a significant decrease of the fracture stress with increasing annealing time was observed.

9:20am **CM2-2-TuM-5 Mechanical Properties of Thin Films Deposited by HiPIMS onto Flexible Substrates**, ***Tereza Kosutova (tereza.kosutova@angstrom.uu.se)***, Uppsala University, Department of Electrical Engineering, Sweden*; M. Tavares da Costa,* Karlstad University, Sweden*; K. Gamstedt,* Uppsala University, Department of Materials Science and Engineering, Sweden*; D. Drozdenko,* Charles University, Czechia*; T. Kubart,* Uppsala University, Department of Electrical Engineering, Sweden

Our study focuses on the strength and ductility of thin films deposited by magnetron sputtering on flexible substrates. Although thin films are widely used in surface engineering, their application on foils brings new requirements on the mechanical properties of the coating material as well as the substrate-coating interface. Here, we aim on the determination of mechanical properties of thin films deposited by dc and high power impulse magnetron sputtering (HiPIMS). The main goal is to identify deposition conditions that ensure good adhesion and ductility of the layers and therefore facilitate applications of coated metal foils.

In-situ testing in an SEM is used to quantify the film cracking during tensile loading and thus analyse the distribution of fracture strain and interfacial shear strength of the coating material. This technique is complemented by the strain field analysis of the substrate foils determined by digital image correlation to identify defects that could induce stress concentration and premature failure. Thin films of copper, titanium and amorphous carbon are evaluated as examples with different intrinsic ductility. Furthermore, the effect of interlayers was investigated. The behaviour of the films deposited on two different foils of aluminium and PET is compared.

To identify the impact of different deposition parameters, we analysed a series of samples deposited using different values of the duty cycle and the substrate bias. The results are correlated to the morphology, microstructure and chemical composition analysed mainly by SEM, XRD and EDX techniques. Whereas the copper exhibits high ductility and a good adhesion can be achieved with an ion assistance, the fracture behaviour of the titanium is dependent on the growth conditions.

9:40am **CM2-2-TuM-6 Fatigue-Induced Abnormal Grain Growth in Metallic Thin Films**, *Q. Li,* Georgia Institute of Technology, USA*; A. Barrios,* Colorado School of Mines, USA*; Y. Yang,* Georgia Institute of Technology, USA*; M. Jain,* Sandia National Laboratories, USA*; Y. Liu,* Georgia Institute of Technology, USA*; B. Boyce,* Sandia National Laboratories, USA*; T. Zhu,* ***Olivier Pierron (olivier.pierron@me.gatech.edu)***, Georgia Institute of Technology, USA

This presentation describes a microelectromechanical system (MEMS) based setup to investigate grain growth in ultrafine grained and nanocrystalline metallic thin films under high/very high cycle loading conditions (i.e., up to 10^9 cycles). The advantage of the technique is that it can test different metals (fcc, bcc, different textures) under identical loading conditions. The governing hypothesis is that abnormal grain growth occurs under this loading regime, and that the family of growing grains is mainly dictated by elastic anisotropy. Our preliminary results on Au and Al thin films are compared to our previous work on ultrafine grained Ni. Abnormal grain growth in Au is observed, as in Ni, however, the orientation of the growing grains is different for Au and Ni, given the difference in initial texture. The experimental results can be compared to micromechanics analyses and phase-field modeling, in order to better understand the origins of the thermodynamic driving force.

10:00am **CM2-2-TuM-7 Nanoscale Fatigue Measurements on Diamond-Like Carbon Coatings**, ***Joshua Vetter (joshua.vetter@de.bosch.com)***, *M. Günther, P. Hofmann, S. Grosse,* Robert Bosch GmbH, Germany*; S. Schmauder,* University of Stuttgart, Germany

Diamond-like carbon (DLC) coatings are frequently used to improve wear performance of technical components in tribological systems. Appropriate fatigue properties of the coating system are fundamental to ensure the functionality. The mechanical failure of the coating can lead to spallation and delamination processes already through further low external stresses and hence to total component failure. Therefore, precise measurements are mandatory. Established analysis like the Rockwell test according to DIN 4856 as well as scratch tests are performed with a single overload above the critical strength of the DLC coating disregarding real operating conditions and failure mechanisms e.g. fatigue through cyclic loads. In our study, we performed cyclic nanoscale fatigue measurements of DLC coatings to consider application-related stresses. The coating systems were deposited by PVD and PACVD techniques varying e.g. the thicknesses of the functional and support layer systems. Cyclic nanoindentation measurements with spherical diamond indenters were performed to evaluate the fatigue behaviour. We have shown that promising results can be obtained from this type of measurements. Suitable test parameters were defined to investigate a wide range of different coating systems within a few hours by adjusting static force and force amplitude. Effects of different coating designs, layer thicknesses and mechanical properties (e.g. indentation hardness, indentation modulus, residual stress and yield strength) of the DLC layer could be evaluated. The critical stress for various DLC layer thickness was evaluated with a FEM simulation and compared to the results obtained from the fatigue measurements. With this, essential adjustments of the mechanical properties of the DLC layer were found to increase the fatigue limit. Furthermore, the effect of an additional pre-treatment by annealing under elevated temperatures from 250 °C up to 450 °C was investigated. The results of the nanoscale fatigue test provide information on previously unknown effects that could not be detected with nanoindentation hardness tests. For various DLC systems the thermal degradation under application related stresses could be shown. Our new measurement technique reveals that previous measurements e.g. adhesion measurement results from typical single overload or hardness measurements tests are not conclusive enough to consider application-related load spectra and that cyclic loads are necessary to guarantee the requested operation condition testing environment. Hence the developed fatigue test allows us to adapt the coating systems to the requirements of the real components.

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