Advanced Characterization, Modelling and Data Science for Coatings and Thin Films
Room Town & Country C - Session CM2-1-ThM

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

Moderators: Matteo Ghidelli, CNRS, France, **David Holec**, Montanuniversität Leoben, Austria

8:00am **CM2-1-ThM-1 Nano-Mechanical Characterization and Modeling of Plasticity in Metallic Materials*, Takahito Ohmura,*** Kyushu University/NIMS, Japan **INVITED**

Plastic deformation behavior is characterized through nano-mechanical testing in a small scale associated with microstructures including inter-phase and grain boundary in metallic materials. Deformation behavior was evaluated for Fe-Si bicrystal with different grain boundary plane with S9{221} and S9{114}1). The resistance to a slip transfer at the grain boundary depends on a combination of crystallographic orientation and dislocation character. Plasticity initiation behavior was characterized for ferrite-cementite interface with different coherency in a pearlitic steel2). The critical stress for the plasticity initiation is lower for a semi-coherent interface than that for an incoherent one, suggesting a potential reason for the continuous yielding phenomenon in macroscopic stress-strain curve of the steel with semi-coherent interface. Transmission Electron Microscope (TEM) in-situ straining was applied to reveal dislocation-grain boundary interactions. In the case of ultra-fine grain steel, dislocations in grain interior can sink at the grain boundary with no remarkable pile-up3). This behavior indicates a dislocation density dominancy for the extra-hardening in the UFG steel. Dislocation-Dislocation interaction was also captured through TEM in-situ straining4). The dislocation reaction forms a stable grain boundary, which could be an elementally step of grain refining during severe plastic deformation. The critical stress for a slip transfer was estimated for □3 boundary of pure Al5). The mechanism of the slip transfer can be modeled in a simple dislocation reaction generating a grain boundary dislocation. Deformation mechanisms of plasticity initiation and subsequent behavior were modeled through stochastic analysis based on a pop-in phenomenon on a loading segment obtained from nanoindentation measurement6). The critical stress for the plasticity initiation shows Gaussian like distribution function, indicating a thermally-activated process including a nucleation of shear loop dislocation at defect-free region. In the subsequent stage, the loading curve shows intermittent plasticity, and the probability function for the event magnitude shows power-law type, suggesting a catastrophic phenomenon with a fractal dimension such as dislocation avalanche.

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8:40am **CM2-1-ThM-3 Accelerating Workflows for High-Throughput Nanoindentation*, Eric Hintsala,*** *Kevin Schmalbach, Douglas Stauffer,* Bruker Nano Surfaces, USA

Heterogenous microstructures are commonly employed across a wide range of applications as a tool for materials scientist to engineer the bulk properties, which can be seen in composite materials, multi-phase alloys or even surface treatments and coatings. Sometimes, property distributions can also arise due to processing history, with laser-based techniques with micro-scale heat affected zones being of particular interest recently.In most cases these structures are nano- to microscale in size, so to isolate mechanical properties from individual regions high-throughput nanoindentation-based techniqueshave become increasingly popular.

Two recent advances in nanoindentation mapping are highlighted here. First, the indentation depth controls the finest spacing that can be utilized without affectingthe subsequent nearby indentations and thereby defines the resolution of a nanoindentation map. Displacement control is particularly important when mapping samples with highly variable hardness. To address this, recent enhancements for the HysitronTI 990 TriboIndenter(Bruker, USA) allows for trigger points to be used to switch segments and feedback control all within one test.This enables ahigh-throughput workflow where translation between positions is followed by approach,surface detection, and a displacement-controlled indent. This mode workswith both high load and low load transducers for addressing a large range of depths and spacings.

Secondly,relating the measured mechanical properties to local structure and composition is also essential for materials development.This can be done by switching instruments from the nanoindenter to the SEM, but this is time consuming and generally necessitatesuse of fiducial markers. To facilitate this process,theHysitron PI 89 Auto (Bruker, USA) in situ SEM indenter utilizes a rotation-tilt stage to move the sample between 3 distinct positions: Indentation position, top-down SEM and EDS position, and 70°tilted EBSD position. The accompanying software enables the same sample region to be co-located in all 3 positions easily, such that regions of interest from an EBSD or EDS map can be directly targeted for indentation testing.

9:00am **CM2-1-ThM-4 Understanding the Fracture Behavior, Interface Characteristics of Micro and Nanocrystalline Diamond Laminates Through Flexural Studies*, Krishna Sarath Kumar Busi,*** Technical University Darmstadt, Germany*; Tim Fuggerer,* University of Erlangen-Nuremberg, Germany*; Sebastian Bruns,* Technical University Darmstadt, Germany*; Timo Fromm, Stefan M Rosiwal,* University of Erlangen-Nuremberg, Germany*; Karsten Durst,* Technical University Darmstadt, Germany

Diamond metallic laminates (DML) have been demonstrated to exhibit an improved toughening mechanism by modifying the crack driving force with alternate hard and ductile layers [1]. These laminates were produced from free-standing diamond foils using HFCVD, exhibiting distinct crystalline morphologies microcrystalline (conventional) and nanocrystalline integrated with metallic layers deposited through PVD. A systematic investigation was set up to understand the mechanical behavior, interface characteristics of these multilayer system through macro 3PB and micro cantilever flexural studies. Nanocrystalline diamond foils exhibited better toughness, and their fracture sensitivity was analyzed by recording continuous stiffness change with respect to crack propagation for notched cantilevers using nanoindentation. Significant delamination was observed in nanocrystalline laminate (nDML) exhibiting weak interfacial strength between diamond and metal layers. Suitable analytical laminate models were effectively applied to investigate shear stress distribution, critical cracking events and the extent of delamination. Additionally, A 2D model of FEM with cohesive interactions was designed in same experimental scenarios (3-point bending, micro cantilever bending) to understand the diamond-metal interfacial properties, showed strong alignment with the analytical models and offered valuable insights to optimize the overall design of the laminate.

Keywords: Laminates, nanoindentation, toughness, fracture, bending, FEM.

References:

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9:20am **CM2-1-ThM-5 Deposition of Hierarchical Ti/Ti_2AlC Metal/MAX Multilayered Nanolaminates and Investigating their Mechanical Properties and Deformation Mechanisms*, Amruta Vaghela,*** Iowa State University, USA*; Skye Supakul,* Pacific Northwest National Laboratory, USA*; Kevin Jacob, Sid Pathak,* Iowa State University, USA

We report the challenges (e.g. the diffusion and formation of MAX phase) with the high temperature synthesis of a hierarchical metal/MAX phase multilayered nanolaminate (MMN) where the interface between metal and MAX phase layers are in direct competition with the internal interfaces within the MAX layers. Using a combinatorial physical vapor deposition (PVD) and atomic layer deposition (ALD) system, we report our first successful deposition of a Ti/ $Ti_{n+1}AlC_n$ (n=1 or 2) MMN where thin Al_2O_3 diffusion barrier layers inhibits interlayer diffusion and enables the multilayered architecture to be retained. The mechanical properties of the MMN system show impressive indentation hardness (12.75±0.33GPa), micro-pillar instability stresses (8.1±0.2GPa), and instability strains (8.3±0.4%) compared to a rule-of-mixtures approximation of the hardness and yield strength of the system. Post compression TEM analysis is also being conducted to gain insight on the deformation mechanisms at play in these hierarchical MMN systems.

9:40am **CM2-1-ThM-6 Effect of Fe Addition on the Structural, Mechanical and Electrical Properties of $(ZrCu)_{1-x}Fe_x$ Thin Film Metallic Glass***, **Evgeniy Boltynjuk**, Yulia Ivanisenko,* KIT, Germany*; Marco Ezequiel, Francesco Bignoli, Damien Faurie, Philippe Djemia, Matteo Ghidelli,* CNRS, France*; Horst Hahn,* Oklahoma State University, USA

Thin film metallic glasses (TFMGs) have emerged as a promising class of materials for applications in the field of flexible electronics, owing to their large deformability, metallic-like electrical conductivity, and low or even negative temperature coefficient of resistivity. However, despite these advantages, the properties of TFMGs require improvement to expand their range of applications. In this study, we focus on $(ZrCu)_{1-x}Fe_x$ system, as the properties of the ZrCu system have been investigated across a broad range of compositions, while the addition of Fe can potentially improve its glass forming ability and thermal stability. Specifically, we will discuss experimental results and *ab initio* calculations on the effect of Fe addition on mechanical and electrical properties, with a focus on clarifying the relationship between these properties and atomic structure and local ordering.

$(Zr_{34}Cu_{66})_{1-x}Fe_x$ TFMGs were synthesized by magnetron sputtering varying the Fe content from 0 up to 76 at.%. All obtained samples, up to the highest Fe content (76 at. %), have an amorphous structure, indicating a high glass forming ability. The electrical resistivity shows a monotonic decrease with increasing Fe content, reducing from 192.4 down to 113.8 μΩ × cm. At the same time, tensile tests on polymeric substrate show a reduction in crack onset strain (COS) from ~ 2.2 down to 1.6% in the range of Fe concentrations from 0 to 54 at.%. For higher Fe concentrations COS shows inverse trend, reaching ~ 2.0% at 76 at.% Fe. Thus, TFMG containing 76 at.% of Fe shows COS value comparable to that of the ZrCu, while achieving a 1.7-fold decrease in electrical resistivity.

Transmission electron microscopy and atom probe tomography reveal structural modifications, mapping the evolution of local ordering and atomic arrangements. By correlating our experimental findings with *ab initio* calculations, we establish a clear relationship between the modification of atomic order and performance of $(ZrCu)_{1-x}Fe_x$ TFMGs. This correlation not only enhances our understanding of TFMGs but also provides a solid foundation for their potential applications in flexible electronics, guiding future research in optimizing TFMGs for advanced technological uses.

10:20am **CM2-1-ThM-8 Mechanical Properties of Thin Films Studied using 4D-STEM***, **Christoph Gammer**,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben, Austria*; Alice Lassnig,* Montanuniversität Leoben, Leoben, Austria*; Lukas Schretter, Simon Fellner,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben, Austria*; Jürgen Eckert,* Montanuniversität Leoben, Leoben, Austria **INVITED**

The mechanical behavior of thin films is highly dependent on their microstructure Micromechanical testing can be used to study small-scale mechanical properties. Modern thin film systems are becoming increasingly complex and their overall mechanical properties are influenced by strong variations in the local elastic and plastic response. Therefore, to understand their deformation behavior the local nanoscale stress distribution during loading has to be considered. The overall load-displacement curve is not sufficient. Recently, we have demonstrated that 4D-STEM allows to perform strain mapping at the nanometer scale during continuous *in situ* deformation in the TEM. In the present talk we will present recent advances demonstrating how 4D-STEM can be used to understand the deformation mechanisms in single-crystalline, nanocrystalline and amorphous thin films.

11:00am **CM2-1-ThM-10 Investigating the Interplay between Biaxial Multicracking of Nanometric Thin Films and Their Magnetic Properties: A Nuanced Separation of Magnetoelastic and Magnetostatic Effects***, Hatem Ben Mahmoud, **Damien Faurie**,* Laboratoire des Sciences des Procédés et des Matériaux (LSPM) – CNRS, France*; Pierre-Olivier Renault, Pierre Godard,* Institut Pprime - CNRS - ENSMA - Université de Poitiers, France*; Dominique Thiaudière, Philippe Joly, Christian Mocuta,* Soleil Synchrotron, France*; Eloi Haltz, Noël Girodon-Boulandet, Fatih Zighem,* Laboratoire des Sciences des Procédés et des Matériaux (LSPM) – CNRS, France

The magnetoelectronic systems of the future will be designed to adapt to complex geometries. Flexible electronics have seen rapid growth, offering promising applications in areas such as confined environments and flexible displays [1]. These systems rely on polymers, which are lighter and more cost-effective than silicon. Understanding the interplay between mechanical strain and magnetic properties is essential [2]: at low strains, magnetic anisotropy is key, while at higher strains, microscopic damage (e.g., fragmentation and decohesion) becomes critical [3].

However, the relationship between thin film fragmentation and magnetic properties [4], especially under biaxial tension, remains poorly studied. This thesis aims to fill this gap through novel experimental techniques and studies on model systems. We investigated flexible magnetic systems using in situ methods, applying significant mechanical strain (up to 10%) while simultaneously probing their magnetic properties. A magneto-optical Kerr effect (MOKE) magnetometer was developed at the DiffAbs beamline of the Soleil synchrotron. Our research focuses on the distribution of stress (before and during cracking) and its impact on the magnetic response, along with the underlying mechanisms.

Two main mechanisms are identified: first, stresses generated during mechanical loading can induce a strong magnetoelastic field that alters the magnetic response; second, magnetostatic fields between fragments separated by cracks can contribute to coercivity during magnetization cycles. However, these effects have yet to be fully quantified. To address this, we studied magnetization cycles under large deformations in Ni80Fe20 films (with negligible magnetoelastic contribution), of varying thickness, with or without W layers of different thicknesses to modify fragment size. We demonstrate that the magnetostatic contribution is closely linked to the aspect ratio (diameter/thickness) of the fragments. This study, compared with research on Co layers, clearly distinguishes between geometric (magnetostatic) effects and stress-induced (magnetoelastic) effects.

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11:20am **CM2-1-ThM-11 Cross-Sectional Nanoindentation Mapping of Sputtered Inconel 725 Films***, **Ikponmwosa Iyinbor**,* Mork Family Department of Chemical Engineering and Materials Science, University of Southern California., USA*; Jin Wang,* Institute of Energy Materials and Devices, Microstructure and Properties of Materials (IMD-1), Forschungszentrum Jülich GmbH., Germany*; Ruth Schwaiger,* Institute of Energy Materials and Devices, Microstructure and Properties of Materials (IMD-1), Forschungszentrum Juelich GmbH., Germany*; Andrea Hodge,* Mork Family Department of Chemical Engineering and Materials Science, University of Southern California., USA

Heterogeneous nanostructured materials (HNMs) offer significant potential for overcoming the strength-ductility trade-off observed in conventional homogeneous nanostructured materials. Recently, we have demonstrated the influence of heterogeneous stress distribution on the development of unique nanostructured features in sputtered nanotwinned Inconel 725 thick films after undergoing heat treatment. A gradient microstructure with three distinct nanodomains featuring a nanocrystalline equiaxed region, a nanotwinned region with carbides, and a region featuring abnormal recrystallization wherein abnormally large grains, delta-phase precipitates, and rafted structures were observed. This unique combination of nanodomains is expected to contribute distinct responses to mechanical deformation behavior.

In this work, two different HNMs of 8 μm and 20 μm film thicknesses were synthesized and heat treated. A nanoindentation mapping technique using a Femto-Tool NMT04 in-situ SEM nanoindenter was performed in order to generate high-spatial resolution hardness and elastic modulus property maps. The nanoindentation maps show a good correlation to the observed heterogeneous microstructure, revealing trends that provide an understanding of the local deformation behavior of these HNMs. Understanding the contribution of each nanodomain to the overall deformation behavior of the films enables the optimization of design and fabrication strategies to provide a superior combination of properties.

11:40am **CM2-1-ThM-12 Fracture Behaviour of Crystalline Metal/Amorphous Oxide Nanolaminates**, ***Thomas Edwards,*** NIMS (National Institute for Materials Science), Japan; *Hendrik Jansen,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland; *Seiichiro II,* NIMS (National Institute for Materials Science), Japan; *Barbara Putz, Johann Michler,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland

The extent of the embrittlement in ductile-brittle multilayers often depends on the modulation period ($t_{brittle}$ + $t_{ductile}$) as well as on the modulation ratio ($t_{brittle}/t_{ductile}$) [1]. Ductile-brittle thin film multilayers of crystalline Al and amorphous AlO_x are studied by our group, produced by atomic layer (AlO_x) and physical vapour deposition (Al) uniquely-combined within a single deposition system. Such multilayer films are thermally stable up to 0.85 T/T_m. Using this ALD/PVD combination, neighbouring layer thicknesses can easily differ by one order of magnitude or more, and both the amorphous oxide layer thickness and that of the metal have been optimised previously for strength (over 1 GPa) and ductility. Here, the effect of this divergence between layers in chemistry, crystallinity and length scale on the fracture behaviour of these thin films was studied by notched microcantilever fracture testing, including at high temperature, and *in situ* TEM tensile loading of notched testpieces to evaluate the role of the amorphous-crystalline interfaces on crack propagation at the nanoscale. The outcomes are compared to those of alternative materials and using other measurement methods.

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