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

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

Moderators: Hanna Bishara, Tel Aviv University, Israel, **Matteo Ghidelli**, Laboratoire des Sciences des Procédés et des Matériaux (LSPM) – CNRS, France

8:20am **CM2-1-ThM-2 Functional Dislocations in Oxides: Near-Surface Engineering and Extension to Thin Films*, Xufei Fang [xufei.fang@kit.edu],*** Karlsruhe Institute of Technology (KIT), Institute for Applied Materials (IAM), Germany **INVITED**

In light of the increasing interest in dislocation-tuned physical properties and the technological potential that dislocations may hold in ceramics, research on dislocations in ceramics is drawing renewed attention. To facilitate the dislocation research in ceramics, the pressing challenge is to engineer dislocations into brittle ceramic materials without inducing cracks. To this end, we have separately examined the dislocation behavior, including dislocation nucleation, multiplication, and motion, enabling us to tailor dislocations into some ceramic materials at room temperature. We can now achieve a dislocation density ranging from ~10^{10}/m² to ~10^{15}/m² in the near-surface region, with a plastic zone size of up to millimeters, using the deformation toolbox developed in our group since 2019. This toolbox allows us to further build on dislocation engineering beyond the near-surface region, going into the thin films and studying the dislocation-tuned mechanical and physical properties. In this talk, I will first introduce the room-temperature dislocation engineering toolbox. Then I will focus on the mechanical and functional properties tuned by the *near-surface dislocations* as well as in the dislocation-engineered thin films. The proofs-of-concept on the model perovskite $SrTiO_3$ will be demonstrated to showcase the applicability.

9:00am **CM2-1-ThM-4 Atomic-Scale Revealing the Mechanical Response of Defect-Mediated Nitride Ceramics*, Zhang Zaoli [zaoli.zhang@oeaw.ac.at],*** Erich Schmid Institute, Austria*; Chen Zhuo, Yong Huang,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben, Austria

Nitride ceramic coating materials exhibit several advantages over metals, including superior hardness, wear resistance, thermal stability, and oxidation resistance [1-3]. With the growing need for industrial applications and environmental considerations, developing new composite nitride coatings that are both economically and environmentally friendly has become a challenging task. Using the architectural structure design of the interface and planar defects could be one approach. Along this line, we made some progress.

The extensive high-resolution transmission electron microscopy (HRTEM) observations of the TaN/TiN multilayer reveal that dissociation of full dislocations results in a network of stacking faults (SFs) and the formation of Lomer-Cottrell lock arrays within the TaN layer. Consequently, the high density of stacking faults dramatically strengthens the TaN/TiN multilayer [1]. Using valence electrons and inner shell electron spectroscopy, a combined experimental analysis of a multilayered structure of CrN/AlN allowed for the mapping of the multilayer's mechanical properties (bulk modulus) at the nanometer scale [2].

We observed atomic-scale intermixing in the nanoscale TiN/AlN multilayer by combining cross-sectional FIB cutting with atomic-resolution electron microscopy. A new solid-solution phase formed, as evidenced by mapping electronic structure differences. Using atomic EDS, we further corroborated that a homogeneous solid-solution zone formed upon loading [3].

From atomic-resolution observations, we first revealed that deformation in vacancy–engineered WNx/TiN multilayers can also be achieved through unit-unit disturbance. Instead of dislocation motion, multiple local unit-cell-scale disturbances can dissipate local strains, thereby releasing stress concentrations and enabling large-scale deformation. This mechanism leads to a significant enhancement of mechanical properties [4]. Moreover, one remarkable advancement is the discovery of an approach that successfully introduces a large density of nanotwins into nitride ceramics [5]. The synergy between the strength and toughness of nitride ceramics is enhanced. [5]

[1] Yong Huang et al., *Acta Materialia* 255 (2023) 119027

[2] Zaoli Zhang et al., *Acta Materialia*, 194(2020) 343

[3] Zhuo Chen et al., *Acta Materialia*, 214(2021)117004.

[4] Zhuo Chen et al., *Nature Communications*, (2023)14:8387

[5] Yong Huang, et al., Acta Materialia 299 (2025) 121475

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9:20am **CM2-1-ThM-5 Probing nanoscale deformation mechanisms in metastable metallic thin films using 4D-STEM*, Lukas Schretter [lukas.schretter@oeaw.ac.at],*** *Jürgen Eckert, Christoph Gammer,* Austrian Academy of Sciences, Austria **INVITED**

Understanding the deformation behavior of metallic thin films at small scales is essential for advancing nanoscale devices and coating performance. Mechanical properties are strongly governed by microstructural features such as grain size, defects, and interfaces, leading to pronounced spatial variations in elastic and plastic response and thus controlling failure. Conventional macroscopic testing is unable to resolve these local effects. In this talk, we present the recent progress in probing the nanoscale deformation mechanisms of metallic thin films at the nanoscale using four-dimensional scanning transmission electron microscopy (4D-STEM). This technique enables in-situ strain and crystal orientation mapping with nanometer spatial resolution during simultaneous mechanical loading inside the transmission electron microscope. Utilizing this advanced characterization technique, we aim to provide quantitative insight into the local strain evolution, stress redistribution, and defect activity that lead to material failure. The results demonstrate how 4D-STEM serves as a powerful tool for linking microstructure and mechanical performance. These insights provide a foundation for designing new material systems with tailored mechanical performance and improved reliability through nanoscale structural design.

10:20am **CM2-1-ThM-8 High-Speed Nanoindentation Mapping and Machine Learning as Enabling Technologies for Combinatorial Thin-Film Libraries*, Edoardo Bemporad [edoardo.bemporad@uniroma3.it],*** Roma tre university, Italy*; Rostislav Daniel,* Montanuniversität Leoben, Leoben, Austria*; Edoardo Rossi,* Roma Tre University, Italy*; Michal Zitek,* Montanuniversität Leoben, Leoben, Austria*; Marco Sebastiani,* Roma Tre University, Italy **INVITED**

Combinatorial thin-film libraries are rapidly transforming the exploration of complex metallic alloys, yet the ability to interpret their mechanical behavior across broad compositional gradients remains a significant challenge. High-speed nanoindentation mapping, combined with advanced data analytics, now provides the statistical depth and spatial resolution required to transform such coatings into quantitative mechanical datasets.

In this study, a compositionally graded Cr–Cu–Ti–W system was synthesized as a model platform to investigate how partial miscibility and non-equilibrium co-sputtering produce diverse architectures: from nanocrystalline solid solutions to amorphous metallic composites. More than 3,000 indents were acquired across 29 regions of interest, establishing position-resolved maps of hardness, modulus, and derived figures of merit (H/E, H^3/E^2). When correlated with local EDX composition and confirmed by STEM-EDS, the results reveal distinct mechanical regimes: Ti- and Cr-rich domains combine strength and compliance, whereas W-enriched regions exhibit high stiffness but limited deformability.

In this framework, unsupervised learning algorithms are applied to analyze the high-speed indentation dataset, identifying clusters of mechanical behavior. These mechanically defined clusters guide targeted investigations into microstructural and micromechanical properties. The analysis utilizes micropillar compression data from over 200 pillars across different regions of interest to directly assess yield strength and strain-hardening behavior.

Unsupervised learning and dimensionality-reduction algorithms classify the pillars based on their deformation responses and connect these classifications to local indentation signatures and transmission electron microscopy (TEM) resolved microstructures. This approach allows for the identification of recurring deformation patterns, such as shear localization, homogeneous flow, or cracking, that are associated with specific compositional and microstructural configurations.

This combined experimental–computational framework provides a pathway for the rational design of multicomponent coatings, in which mechanical functionality emerges from quantitative correlations across scales.

11:00am **CM2-1-ThM-10 Towards Thermal Stability of Interface-Engineered Crystalline / Amorphous Al100-zNiz / AlOxHy Nanolaminates***, Hendrik Jansen [hendrik.jansen@empa.ch], Amit Sharma, Barbara Putz,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland*; Marcus Hans, Jochen Schneider,* RWTH Aachen University, Germany*; Thomas Edwards,* NIMS (National Institute for Materials Science), Japan*; Jakob Schwiedrzik,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland*; Johann Michler,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland

Recent deposition advances allow interface-engineered $Al_{100-z}Ni_z$ / AlO_xH_y (z = 0, 2, and 5 at.%; 25 / 1 nm) nanolaminated thin films by a combination of physical vapour and atomic layer deposition. These enable tailored Al grain boundary engineering in both in-plane and out-of-plane orientation through the precipitation of secondary Ni-rich phases and thermally stable amorphous interlayers. After vacuum annealing between 160°C and 600°C for 1 h, micromechanical testing was complemented by transmission electron microscopy and atom probe tomography to establish the microstructure-property relationship. At room temperature, the films exhibit sub-10 nm lateral Al(-Ni) grain size, smooth crystalline / amorphous interfaces, and Ni-rich nanoclusters – yielding more than 5 GPa of nanoindentation hardness and 2 GPa yield strength in micropillar compression. While microstructural stabilization was achieved at low homologous temperature, different temperature-dependent mechanisms were identified constraining the thermal stability: competing Ni interface decoration and Al_3Ni phase separation, AlO_xH_y crystallization promoted by Ni and H, and eventual interlayer breaking. Overall, these results highlight not only the importance of refining crystalline–amorphous nanolaminate architectures as structural material templates but also demonstrate the potential to monitor hydrogen-related interactions towards barrier coatings and micro-electro-mechanical systems.

11:20am **CM2-1-ThM-11 From Mechanics to Electronics: Influence of ALD Interlayers on the Multiaxial Electro-Mechanical Behavior of Metal-Oxide Bilayers***, **Johanna Byloff [johanna.byloff@empa.ch]**,* ETH Zurich, Switzerland*; Vivek Devulapalli, Daniele Casari,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland*; Thomas Edwards,* NIMS (National Institute for Materials Science), Japan*; Claus Trost, Megan Cordill,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben, Austria*; Shuhel Husain,* Universite Sorbonne Paris Nord, France*; Pierre-Olivier Renault,* Universite de Poitiers-CNRS, France*; Damien Faurie,* Universite Sorbonne Paris Nord, France*; Barbara Putz,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland

Metal thin films on polymers are vital for flexible electronics and space technology, such as in the James Webb telescope sunshield [1] and BepiColombo's multilayer insulation [2], yet their applicability is fundamentally limited by the trade-off between mechanical and functional performance. We show how atomic layer deposited (ALD) amorphous AlO_xH_y interlayers (0–25 nm) directly control the electro-mechanical functionality of sputter-deposited bilayer aluminum films on polyimide substrates using a combined ALD-PVD chamber with in-vacuo transfer for deposition. We utilize multiaxial tensile testing with in-situ film stress and electrical resistance measurements at synchrotron SOLEIL [3] to correlate stress-strain behavior, full width at half maximum (FWHM) evolution and crack onset strains (COS). It is demonstrated that interlayer thickness determines both deformation mechanisms and electrical strain limits. Adding an ALD interlayer between polymer substrate and metal thin film significantly improves the deformability of the entire system. However, interlayer thickness determines both deformation behavior and electric strain limits in a non-linear manner. While a single ALD cycle enhances ductility, surprisingly a 1 nm oxide interlayer between metal thin film and polymer substrate causes early electrical failure. Optimal performance—improved ductility, delayed cracking, and maintained electrical conductivity under large deformation—is achieved at 5-10 nm interlayer thickness. Beyond this range, embrittlement causes early electrical failure through oxide-initiated cracking. PVD film properties can be positively influenced by a preceding ALD step, improving functional performance of the whole system. We show that FWHM evolution is a footprint of ductile or brittle deformation behavior. We establish the margin between mechanical and electrical failure criteria (σ_y and COS) as a quantitative metric of ductility. Critically, we demonstrate that bilayer systems preserve ductility during deformation when the modulation ratio ($t_{brittle}/t_{ductile}$) remains below 0.1, establishing a quantitative design criterion for engineering mechanically robust multilayer coatings. Beyond immediate applications in flexible electronics and space technologies, our findings provide fundamental insights into how competing deformation mechanisms at heterointerfaces can be leveraged to create materials with programmable mechanical and electrical responses.

[1] J. Moery, Space Telescopes and Instr. I, 6265 (2006)

[2] M. Moser et al., High temp. MLI for the BepiColombo spacecraft, ICES (2010)

[3] G. Geandier et al., Rev. Sci. Instrum. 81, 103903 (2010)

11:40am **CM2-1-ThM-12 Deformation Twins, Kink Bands and Stacking Faults: Highlighting the Diversity and Complementarity of Deformation Mechanisms in the MAX Phase Cr_2AlC Through Micromechanical Testing***, **Christophe TROMAS [christophe.tromas@univ-poitiers.fr]**, Mohamed AKOU,* Institut Pprime - CNRS - ENSMA - Université de Poitiers, France*; Salomé PARENT,* institut pprime - CNRS - ENSMA - Université de Poitiers, France*; Anne JOULAIN,* Institut Pprime - CNRS - ENSMA - Université de Poitiers, France **INVITED**

In the process of determining the elementary mechanisms of plastic deformation, micromechanical testing has opened up a new avenue. Nanoindentation testing induces plasticity into a micrometer size volume, providing a localized plastic deformation structure that is easy to observe and identify. A spherical tip, instead of a classical pyramidal tip, avoid stress concentrations and produces a long-range stress gradient, with regions in tension and others in compression or shear, providing a broad sample of the possible mechanism in a given area. Complementarily, compression tests performed using a nanoindenter, equipped with a flat punch, on micrometer-sized pillars prepared by focused ion beam (FIB), generate a uniaxial and uniform compressive stress, easier to analyze. Furthermore, thanks to in situ experiments, observation of the free surfaces of the pillars under compression provides dynamic information on the deformation process.

In this study, the plastic deformation mechanisms of the MAX phase Cr_2AlC (a nanolamellar material with a hexagonal crystallographic structure) is investigated using micropillars compressions experiments and spherical nanoindentation. In both cases, the deformation microstructure is analyzed by Transmission Electron Microscopy (TEM) on lamella extracted along different orientations, in combination with surface observation by Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM), and with local crystallographic misorientation maps (ACOM ASTAR). This approach allows us to study the role played by deformation twinning, kink bands and stacking faults in the plastic deformation processes in this material.

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