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

8:40am **CM2-1-ThM-3 Ultra–High Vacuum Tribology: Industrial Relevance, Mechanisms, and Research Gaps***, **Esteban Broitman [ebroitm@hotmail.com]**, Sven Kelling, Rickmer Kose,* Sentys Inc., USA

Tribological behavior—friction, wear, and adhesion—depends critically on the local environment at contacting surfaces. In ambient air, adsorbed water, oxygen, and organic contaminants form boundary films that dominate contact mechanics and chemistry; as pressure is reduced these physisorbed layers thin and desorb, shifting surface interactions toward intrinsic solid–solid processes. Ultra–high vacuum (UHV), commonly defined as pressures below 10^{-9} mbar, represents an extreme limit in which physisorbed monolayers are effectively absent on laboratory timescales and surface chemistry is governed by atomic–scale adsorption and chemisorption. UHV conditions therefore provide a unique window onto fundamental friction and wear mechanisms that are masked at higher pressures.

For industrial applications, UHV tribology is directly relevant to sectors where components operate in extreme vacuum or require contamination–free contacts: satellite mechanisms and deployable structures, scientific instruments and space optics, semiconductor and thin–film processing tools, electron– and ion–beam systems, particle accelerators, and vacuum–operated MEMS/NEMS. Despite this industrial relevance, UHV tribology remains comparatively rare: most experimental work is performed in atmosphere or in high vacuum (HV, 10^{-3}–10^{-7} mbar), where residual gases and humidity continue to influence outcomes. The scarcity of UHV studies reflects practical barriers—specialized chambers, rigorous sample preparation and bakeout, vacuum–compatible instrumentation, and long pumpdown cycles—as well as a perception that UHV results have limited applicability to real–world service. Commercial UHV tribometry options are extremely limited; **PREVAC** currently offers a commercial UHV tribometer capable of reaching pressures on the order of 10^{-9} mbar, representing one of the few turnkey solutions for routine industrial UHV tribological testing.

This presentation evaluates UHV tribology through an industrial lens, bridging the gap between fundamental research and practical application. By comparing friction and wear data across UHV, high vacuum, and atmospheric conditions for common materials and coatings, we identify critical performance shifts. We conclude with actionable design recommendations aimed at accelerating the integration of UHV tribology into industrial hardware for product design and development.

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 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.

11:40am **CM2-1-ThM-12 Analysis of the Mechanical Properties of APS Coatings Deposited on Agricultural Plough Components***,* *Boris Nazar,* Technical University of Moldova*; Fabian Cezar Lupu, Corneliu Munteanu, Viorel Goanta,* ***Bogdan Istrate [bogdan.istrate@academic.tuiasi.ro]****,* "Gheorghe Asachi" Technical University of Iasi, Romania*; Grigore Marian,* Technical University of Moldova*; Marcelin Benchea,* "Gheorghe Asachi" Technical University of Iasi, Romania

The present paper presents research conducted in the field of thermal spray coatings aimed at improving the properties of agricultural components. The studies focus on thermal plasma jet deposits using the APS (Atmospheric Plasma Spray) technique applied to agricultural plough components designed for soil processing. These components are subjected to extreme operating conditions during use, and their main required properties are wear and impact resistance - key performance factors that determine the plough's service life.

In the research, thermal coatings were produced using WC–12%Co–based metallic powders (commercial name WOKA 3101). On laboratory samples, mechanical properties were evaluated through tensile tests, micro-scratch testing, and determination of the coefficient of friction under both rotational and translational motion. Since these components experience significant operational stress, thermal spraying represents an effective method not only for improving the mechanical properties of newly manufactured parts but also for refurbishing worn elements to restore them to proper working condition.

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12:00pm **CM2-1-ThM-13 Thin Film Characterization by Ultrasonically Induced Nanofatigue During Nanoindentation***,* ***Antanas Daugela [info@nanometronix.com]****,* Nanometronix LLC, USA

In the era of fast product development thin film researchers are looking for fast and efficient methods of characterization.This is especially true in a semi-conductor industry where advanced multilayered chip/MEMS development process needs advanced characterization techniques. Nanoindentation based multi-cycle loading is offering insides into the real-time contact fracture dynamics [1]. A nanofatigue phenomenon can be observed on thin sub-micrometer films by monitoring the resulting multi-cycle nanoindentation loading-unloading curves where post-test imaging helps in identifying materials' behavior [2, 3].In addition, classical Mason-Coffin and ratcheting fatigue models derived for the nanoscale contact can be utilized in the predictions and correlate reasonably well with nanofatigue cycles obtained experimentally.

A newly developed ultrasonic nanoindentation tip operates at hundreds of kHz, therefore, inducing millions of load cycles within seconds.The resulting nanofatigue induces different thin film fracture modes such as radial, sink-in and produce unique acoustic signatures.The ultrasonic nanoindentation tip monitors associated waveforms, which can provide additional insides into nanofatigue process dynamics via advanced acoustic waveform analysis.Following our previous study [4], acoustic waveforms were processed using a combination of harmonics and Gauss noise base functions for signal decomposition. The proposed Deep Learning technique yields in reliable classification of acoustic signatures obtained during fracturing ofsub-micrometer thick coatings.Multiple SiC and GaAr semi-conductor thin films were tested.

References:

1. B. D. Beake et al, *Materials Science & Engineering A*, 7**80** 139159 (2020)
2. H. Kutomi et al, *Tribology International*, **36**, p.255-259 (2003)
3. Y. Matsuda et al, Wear, **259**, p. 1497–1501 (2005)
4. A. Daugela et al, *Thin Solid Films,* **788**, 140177 (2024)

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