Protective and High-temperature Coatings
Room Town & Country A - Session MA2-2-WeM
Hard and Nanostructured Coatings II

Moderators: Stanislav Haviar, University of West Bohemia, Czechia, **Kuan-Che Lan**, National Tsing Hua University, Taiwan , **Norma Salvadores Farran**, TU Wien, Austria

8:00am **MA2-2-WeM-1 Dual-Phase Crystalline-Amorphous Coatings Based on Thin-Film Metallic Glasses: Synthesis and Properties**,
Petr Zeman [zemanp@kfy.zcu.cz], University of West Bohemia, Czechia **INVITED**

Magnetron sputter deposition has been demonstrated to be a suitable technique for synthesizing metallic glasses as thin films (TFMGs). Thanks to the non-equilibrium conditions of low-temperature plasma and extremely high cooling rates at the atomic scale on the substrate, TFMGs can be prepared with a much wider composition variety and solubility than bulk metallic glasses (BMGs). Moreover, TFMGs exhibit properties and characteristics that surpass those of BMGs as well as conventional metallic and ceramic coatings, particularly in achieving an optimized balance between ductility and strength.

The amorphous structure of TFMGs, characterized by short- and medium-range atomic ordering, combined with their exceptional properties, offers opportunities to create dual-phase architectures incorporating both TFMGs and crystalline materials. These architectures have the potential to overcome the limitations inherent to each constituent phase while enhancing existing properties or even enabling novel functionalities through synergistic phase interactions.

Dual-phase crystalline-amorphous coatings based on TFMGs can be relatively easily prepared in multilayer architectures comprising alternating crystalline and TFMG sublayers. We demonstrated this concept with multilayer Zr-Cu-N coatings consisting of hard ceramic ZrN and ductile glassy ZrCu sublayers. The coatings exhibited enhanced damage tolerance due to effective crack deflection at sublayer interfaces, yielding superior fracture stress and toughness values. Incorporating ZrN-Cu nanocomposite surface sublayers further imparted antibacterial functionality, expanding their potential applications.

The formation of dual-phase crystalline-amorphous coatings based on TFMGs in a nanocomposite architecture presents significant challenges. However, we successfully synthesized such coatings in the Zr-Cu-N and Zr-Cu-B systems using a one-step process of reactive and non-reactive magnetron co-sputtering, respectively. The coatings prepared under optimized conditions were nanocomposites comprising nanocrystalline ZrN or ZrB_2 and glassy ZrCu phases, representing a novel class of nanocomposite coatings combining ceramic and TFMG phases.

The talk will detail the compositional design, synthesis, microstructural evolution, and structure-property relationships of these coatings. Results from ab initio simulations that complement the experimental findings will also be presented, and key differences between the two coating systems will be discussed. It will be shown that these coatings offer promising potential for applications requiring a balance of hardness, toughness, and durability.

9:00am **MA2-2-WeM-4 Hardness and Fracture Toughness Enhancement in Non-Stoichiometric Diboride Superlattices**,
Marek Vidiš [marek.vidis@fmph.uniba.sk], *Tomáš Fiantok, Martin Truchlý, Vitalii Izai, Leonid Satrapinskyy, Tomáš Roch,* Comenius University Bratislava, Slovakia*; Rainer Hahn, Helmut Riedl,* TU Wien, Austria*; Peter Švec,* Slovak Academy of Sciences, Slovakia*; Viktor Šroba, Marián Mikula,* Comenius University Bratislava, Slovakia

Superlattice architecture presents a promising strategy for the simultaneous enhancement of hardness and fracture toughness in hard ceramic films. We demonstrate the success of this approach in transition metal diboride films and report the structural and mechanical properties of films composed of nanocrystalline $ZrB_{2+x}$and disordered TaB_{2-y} layers. Superlattice films with a wide range of bilayer periods (Λ = 1.8–31.5 nm) were prepared by magnetron sputtering. Deposition was performed at 300 °C with a floating bias to minimize interdiffusion. The formation of sharp interfaces for all Λ values is confirmed by X-ray reflectivity. The films consist of strongly understoichiometric $TaB_{1.4}$ layers, which lack long-range ordering, and overstochiometric $ZrB_{2.6}$ layers with a preferential (001) crystalline orientation. With decreasing Λ, we observe a change in preferential orientation and the formation of a true superlattice structure, evidenced by satellite peaks. This indicates crystallization of the $TaB_{1.4}$ layers, as confirmed by STEM data which shows both layers exhibiting a (001)-oriented hexagonal structure. This is a result of two effects: locally induced stabilization by the underlying ZrB_{2+x}layer and boron diffusion at the interface, enhanced by the boron concentration gradient and the bombardment of Ar neutrals reflected from the targets. This transition is accompanied by a remarkable increase in hardness from 34.1 ± 1.9 to 47.2 ± 2.3 GPa as Λ decreases to 3.4 nm. The observed hardening exceeds estimations based on Koehler's strengthening mechanism for two layers with a shear modulus difference of only 39 GPa. Improved mechanical properties are observed also from DFT calculations for defect-free ZrB_2/TaB_2 cells (Λ = 1.4–8.2 nm), which reveal a stabilizing effect with decreasing Λ and a significant increase in stiffness, peaking at Λ = 2.7 nm. At the same time, the fracture toughness K_{IC}, obtained from notched cantilever bending tests, increases from 3.3 ± 0.2 MPa·m$^{1/2}$ (average of both monolithic films) to 4.6 ± 0.3 MPa·m$^{1/2}$ for the superlattice film with Λ = 1.8 nm. This improvement is attributed to coherent stresses at the interfaces due to lattice mismatch. The suppression of brittle response under mechanical load is also confirmed by cube-corner indents, which show shorter radial cracks with decreasing Λ. This work demonstrates that the superlattice approach is highly effective in transition-metal diborides and highlights the crucial role of stoichiometry. It was supported by the Slovak Research and Development Agency (Grant No. APVV-21-0042 and APVV-24-0038), Scientific Grant Agency (Grant No. VEGA 1/0473/24) and COLOSSE project (No. 101158464).

9:20am **MA2-2-WeM-5 Effects of Nitrogen Flow Rate and Deposition Temperature on the Structure and Properties of VMoN Thin Films Deposited by High Power Impulse Magnetron Sputtering**,
Jia-Hong Huang [jhhuang@ess.nthu.edu.tw], *Pei-Fen Peng,* National Tsing Hua University, Taiwan

In this study, vanadium molybdenum nitride (VMoN) thin films were deposited on Si substrate using high power impulse magnetron sputtering (HiPIMS). The purpose of this research was to investigate the effects of process parameters including nitrogen flow rate (N-series) and deposition temperature (T-series) on the structure and properties of VMoN thin films. The results showed that for the coatings deposited at 400 °C, the lattice parameters linearly increased with increasing N/metal ratio, while those deposited at temperatures ranging from 200 to 350 °C, did not follow the Vegard's law. The texture of the VMoN films also significantly affected by the two process parameters. VMoN thin films deposited at 400 °C exhibited a (200) texture, and the texture coefficient of (200) increased with nitrogen flow rate, which could be explained by the steering effect and competitive growth theory. As the deposition temperature decreased, insufficient energy was delivered to the adatoms and promoted the growth of (111)-orientated grains. For the coatings deposited at 400 °C, the ion peening effect became intense with increasing nitrogen flow rate and thereby increasing electrical resistivity from 141.4 to 178.8 μΩ·cm. Furthermore, with increasing N/metal ratio, the hardness of N-series specimens decreased from 25.9 to 17.9 GPa, and compressive residual stress decreased from -3.66 to -1.43 GPa due to the decrease of nitrogen-vacancy hardening effect. In contrast, coatings deposited at temperature ranging from 200 to 350 °C showed no significant variation in N/metal ratio, indicating that nitrogen-vacancy hardening effect was not the primary factor that affected hardness and residual stress of T-series specimens. The results of X-ray diffraction confirmed the presence of a second phase at 350 °C and below, where the resistivity of the specimens substantially increased. The fraction of the second phase increased as deposition temperature decreased, which was correlated with increasing hardness and residual stress. The second phase may play a major role in influencing the properties of T-series specimens.

9:40am **MA2-2-WeM-6 Multi-Scale Investigation of Superior Mechanical Properties in Nitride Ceramics with Negative Stacking Fault Energy**,
Yong Huang, Zhuo Chen, ***Zaoli Zhang [Zaoli.Zhang@oeaw.ac.at]***, Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben, Austria

Introduction: Ceramics are widely used in various structural and functional applications; however, their intrinsic brittleness at room temperature remains a critical challenge, often leading to early-stage catastrophic failures. This brittleness arises primarily due to the high critical-resolved shear stress required to initiate dislocation movement and the limited number of operational slip systems. Addressing this limitation is crucial for the development of ceramics with improved mechanical properties.This study aims to develop a novel strategy for enhancing the deformability of ceramics by leveraging negative stacking fault energy (SFE). The approach seeks to reduce the energetic barriers to dislocation motion and expand the number of available slip systems, ultimately improving room-temperature plasticity while maintaining high strength and toughness. In this work,a TiN/TaN superlattice was fabricated and subjected to in-situ micro-mechanical testing to evaluate its mechanical response. Post-mortem transmission electron microscopy (TEM) was employed to analyze deformation mechanisms at the atomic scale, providing insights into the role of negative SFE in promoting dislocation activity, atomic plane faulting, and twinning.The TiN/TaN superlattice exhibited remarkable room-temperature compressive plasticity (~43%), attributed to extensive atomic plane faulting and twinning facilitated by negative SFE. This behavior enabled an exceptional combination of plasticity, strength, and toughness, demonstrating the feasibility of overcoming the brittleness barrier in ceramics.

References: Huang, Y., et al. (2025). "Harvesting superior intrinsic plasticity in nitride ceramics with negative stacking fault energy." Acta Materialia: 120774.

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11:00am **MA2-2-WeM-10 TiNbN / AlTiNbSiN / CrN Multilayer Coatings Irradiated by 300 keV Ar^+ Ions: The Role of Nitrogen***, Kuan-Che Lan* ***[kclan@mx.nthu.edu.tw]****, Chun-Hung Hsiao,* National Tsing Hua University, Taiwan*; Yin-Yu Chang,* National Formosa University, Taiwan

To study the crystalline stability of nitride coating against heavy ion irradiation for nuclear-related applications,nitride multilayer coatings with the architecture of TiNbN/AlTiNbSiN/CrN of an average period of 25 nm deposited by cathodic arc deposition were irradiated with 300 keV Ar^+ ions at the initial of room temperature under vacuum to the damage levels upto 7 dpa (displacements per atom). The irradiation-induced the change in crystalline structure, composition, and mechanical properties and electrical properties were systematically investigated. Ar^+ ions irradiation, noticeable interdiffusion between adjacent layers was observed. The region of depth exhibited a decrease in nitrogen content after irradiation which is consistent with the prediction of SRIM simulation. The reduction in hardness, electrical conductivity of the coating has been monitored. Besides, post irradiation examination of TEM and APT were carried out to investigate the depth distribution of irradiation induced defects at atomic level resolution. The defects of the role of nitrogen behavior of the properties among a variety of nitride with in the multilayer coating.

11:20am **MA2-2-WeM-11 Extremely Versatile Coating Design Through Adjustable Magnetic Field Settings for Arc Sources Using the Advanced Arc Technology from Oerlikon Balzers***, Alexandre Michau* ***[alexandre.michau@oerlikon.com]****, Denis Kurapov,* Oerlikon Surface Solution AG, Liechtenstein

Magnetic fields play a critical role in cathodic arc deposition processes. Usually applied in the vicinity of the targets, they steer the arc spots and have a big impact on the plasma and subsequent coating properties [1-2]. While random arc motion might be desirable for specific applications, modern processes rely on steered arc motions because it significantly reduces the number of macroparticles incorporated in the coatings [3]. Few studies address magnetic field configurations because they are typically way more complex to implement and modulate compared to other conventional deposition parameters like temperature, pressure or bias voltage.

The optimal magnetic field depends on the coating material to evaporate and grow as well as on its targeted properties and performance. However, such versatility is rarely available at an industrial scale. The Advanced Arc Technology (AAT) from Oerlikon Balzers offers unprecedented capabilities for precise magnetic field tuning and thus versatile coating design in combination with high efficiency of deposition process as well as reduced surface roughness.

The maximum achievable magnetic field density is now twice as high as before, opening a new process window for target materials that are sensitive to high steering speeds. Furthermore, the magnetic field dynamicity, in other terms the speed at which the magnetic field can be changed, approaches the ms scale. This enables advanced strategies such as nanolayering with alternating antagonistic magnetic configurations, opening a new coating architecture window. Finally, the magnetic field flexibility has been improved, allowing its shape and intensity to match the ones from previous generations while introducing new features such as a controlled discharge voltage offering gain in reproducibility and operational simplification.

We will discuss the possibility to tune coating properties with different magnetic field configurations generated using the Advanced Arc Technology. The focus is be given on the magnetic field density (parallel and orthogonal components compared to the target surface) while working with different focused shapes. The versatility of the coating design is demonstrated using two material systems. (Al,Cr)N and (Al,Ti)N are deposited in an industrial scale coater (INVENTA kila from Oerlikon Balzers) using the reactive mode of the following metallic targets: AlCr targets with Al ≥ 70 at.% and AlTi targets with Al ≥ 67 at.%.

[1] A.B.B Chaar et al, Coatings, 9 (2019) 660.

[2] F.F. Klimashin et al., Materials and Design, 237 (2024) 112553.

[3] P.D. Swift, Journal of Physics D: Applied Physics, 29 (1996) 2025.

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