

## Protective and High-temperature Coatings Room Palm 3-4 - Session MA3-3-WeM

### Hard and Nanostructured Coatings III

**Moderators:** Rainer Hahn, TU Wien, Institute of Materials Science and Technology, Austria, Stanislav Haviar, University of West Bohemia, Czechia, Fan-Yi Ouyang, National Tsing Hua University, Taiwan

8:00am **MA3-3-WeM-1 Controlling Phase Selection, Preferred Orientation, and Van Der Waals or Conventional Epitaxy in Molybdenum Oxide Films,** *Faezeh Alijan Farzad Lahiji*, Linköping University, IFM, Sweden; *Biplab Paul*, PLATIT AG, Switzerland; *Ganpati Ramanath*, Rensselaer Polytechnic Institute, USA; *Arnaud le Febvrier*, *Per Eklund*, Uppsala University, Angstrom Laboratory, Sweden

Molybdenum oxide  $\text{MoO}_x$  films ( $x = 2, 3$ ) are attractive for a variety of applications, but exclusive phase selection remains challenging due to the presence of multiple polymorphs. To achieve pure van der Waals epitaxy (vdWE), which relies on van der Waals forces for strain-free growth, we explored phase control of molybdenum oxide ( $\text{MoO}_x$ ), aiming to grow both non-layered monoclinic  $\text{MoO}_2$  and layered orthorhombic  $\alpha\text{-MoO}_3$ .

By adjusting the oxygen pressure  $p_{\text{O}_2}$  during reactive magnetron sputtering, we demonstrate control over phase selection, texture, and epitaxy in  $\text{MoO}_x$  films on f-mica and c-sapphire. Our results show non-layered monoclinic  $\text{MoO}_2$  formation on both f-mica and c-sapphire at 500 °C for  $0.1 \leq p_{\text{O}_2} \leq 0.25$ , outside which the films are amorphous. The result is similar at 400 °C except for layered orthorhombic  $\alpha\text{-MoO}_3$  formation at high  $p_{\text{O}_2}$  and a greater sensitivity of phase selection to  $p_{\text{O}_2}$ . High  $p_{\text{O}_2}$  fosters large thin-sheet crystals with texture and/or epitaxy, while low  $p_{\text{O}_2}$  results in fine-grained flowery microstructure. Pole figure analyses reveal that the orthorhombic  $\alpha\text{-MoO}_3$  films on f-mica exhibit van der Waals epitaxy [1] wherein the out-of-plane  $0k0$  d-spacings are invariant with film thickness, indicative of negligible interfacial strain. In contrast,  $\alpha\text{-MoO}_3$  films on c-sapphire showed  $0k0$  fiber texture. In the case of  $\text{MoO}_2$ , epitaxy occurs on both f-mica and c-sapphire characterized by conventional epitaxy with strong interfacial bonding, promoting strain in the film but enables high crystalline quality and controlled orientation on both substrates [2]. The aforementioned results provide a framework for the controlled synthesis of  $\text{MoO}_x$  films with tunable orientation, epitaxy, strain, and microstructure for applications.

[1] E. Ekström, *et al.*, *Materials & Design* 229 (2023) 111 864.

[2] F.A. Lahiji, *et al.*, *Thin Solid Films* (2024) 140566.

8:20am **MA3-3-WeM-2 Comparative Study of the Effect of W and Nb Addition on Microstructure and Properties of Zr-Cu-Based Thin-Film Metallic Glasses,** *Deepika Thakur*, *Michaela Červená*, *Radomír Čerstvý*, *Petr Zeman*, University of West Bohemia - NTIS, Czechia

Zr-Cu-based thin-film metallic glasses (TFMGs) have emerged as a promising class of materials due to their exceptional properties such as high glass-forming ability, superior elastic strain limit, enhanced hardness and plasticity. Moreover, these TFMGs offer the potential to be combined with nanocrystalline materials (transition metals or metal nitrides) to create heterogeneous dual-phase nanocomposite structures and thus achieving a better balance of toughness and hardness and/or unlocking new functionalities.

Therefore, this study explores the effect of gradual addition of W (negative mixing enthalpy with Zr but positive with Cu) and Nb (positive mixing enthalpy with both Zr and Cu) on microstructure and properties of Zr-Cu-based TFMGs. Two film series, W-Zr-Cu and Nb-Zr-Cu, were prepared, keeping Zr:Cu as 1:1 and gradually varying the W and Nb content in the respective series. Each deposition was done in Ar using three magnetrons equipped with Zr and W/Nb targets operated in the dc regime and a Cu target in the HiPIMS regime.

A systematic investigation revealed that W and Nb additions have a significant impact on microstructure and other properties of the films. The films remain amorphous with smooth surfaces (roughness < 2 nm) up to 65 at.% of W or Nb, displaying vein-like features typical of metallic glasses upon fracture. W-Zr-Cu films with 67 at.% W are characterized by a combination of featureless structures (amorphous-like) close to the substrate and thin columns in the upper part of the film. Films with even higher W contents grow in a V-shaped columnar microstructure corresponding to the bcc  $\alpha\text{-W}$  crystalline structure. Nb-Zr-Cu films with 70 at.% Nb clearly exhibit a dual-phase structure with thin columns

surrounded by vein-like features. Further increase in the Nb content above 70 at.% leads to the formation of a crystalline structure with parallel columns and very small voids. These voids tend to vanish with increasing Nb content. A gradual increase in hardness and reduced Young's modulus is observed with increasing W content for the amorphous W-Zr-Cu films and the crystalline films show an enhancement in hardness of up to 15% compared to films with pure W due to solid solution hardening. In the case of Nb-Zr-Cu films with up to 70 at.% Nb, the hardness remains nearly constant. However, further addition of Nb results in a decreased hardness and this reduction might be attributed to a less dense structure of the films.

Results of ongoing analysis and experiments on W-Zr-Cu and Nb-Zr-Cu films based on ZrCu TFMG will also be presented, providing new insights into the material's phase transitions, mechanical strength, and electrical properties.

8:40am **MA3-3-WeM-3 Tailoring Nanostructure and Functional Properties of Sputter-Deposited Cu-Based Films by Zr Alloying,** *Mariia Zhadko*, *Anna Benediktová*, *Radomír Čerstvý*, *Jiří Houška*, *Jiří Čapek*, *David Kolenatý*, *Pavel Baroch*, *Petr Zeman*, University of West Bohemia, Czechia

Cu and Cu-based films, known for their superior electrical and thermal conductivity, find primary applications in electronic devices and the electrical industry. However, the implementation of various strengthening mechanisms often compromises the conductivity. Therefore, it is crucial to carefully control the structural state and composition of these films to achieve an optimal balance between mechanical strength and conductivity.

In this work, we prepared nanocrystalline Cu-Zr films with a minor Zr content ranging from 0 to 2.7 at.% using non-reactive direct current magnetron co-sputtering of separate Cu and Zr targets in pure Ar at a pressure of  $\sim 0.5$  Pa without substrate bias and external heating. The effects of Zr alloying on the structure, surface, mechanical, and electrical properties were systematically investigated using X-ray diffraction, electron microscopy, atomic force microscopy, indentation, and the four-point probe method. We demonstrate that Zr alloying within the investigated composition range is an effective approach for modifying the structural state and properties of sputter-deposited films, with the most notable changes observed between 0.3 and 1.3 at.% Zr. Beyond this range, only minor changes in the microstructure and mechanical properties are observed, while the solubility, electrical resistivity, and surface roughness continue to rise.

Our systematic investigation shows that during film deposition, a redistribution of Zr atoms occurs between the supersaturated solid solution and grain boundaries resulting in the formation of a complex microstructure along with significant texture weakening and structural refinement. As a result, the alloyed Cu-Zr films exhibit hardness values between 3.2 and 4.2 GPa, exceeding the 2.5 GPa observed in the unalloyed Cu film. This hardness enhancement is attributed to the combined effect of grain boundary strengthening due to the structural refinement and Zr segregation, and solid solution strengthening. An observed increase in electrical resistivity is primarily attributed to electron scattering by Zr atoms dissolved in the Cu lattice and additional scattering at the grain boundaries, especially at Zr contents above 1.5 at.%. However, the as-deposited Cu-Zr films exhibit a combination of hardness and electrical conductivity that is comparable to or better than reported values in the literature. These findings provide a pathway for optimizing structure-property combinations in Cu-Zr films and suggest potential for further enhancement of mechanical and electrical properties through the precipitation hardening mechanism.

9:00am **MA3-3-WeM-4 Influence of Bilayer Periodic Thickness Ratios on the Mechanical Properties and Corrosion Resistance of AlCrNbSiTiN/AlCrN High-Entropy Alloy Nitride Multilayer Thin Films,** *Shang-Hua Tseng*, National Taiwan University of Science and Technology, Taiwan; *Jyh-Wei Lee*, Ming Chi University of Technology, Taiwan; *Chaur-Jeng Wang*, National Taiwan University of Science and Technology, Taiwan; *Bih-Show Lou*, Chang Gung University, Taiwan

High entropy alloy (HEA) nitride thin films have attracted considerable attention from the global industrial and academic communities due to their excellent mechanical properties. HEA multilayer nitride films also exhibit good interfacial stability, outstanding mechanical performance, and superior corrosion resistance. In this study, AlCrNbSiTiN/AlCrN nitride multilayer thin films were deposited using a high power impulse magnetron sputtering (HiPIMS) system with AlCrNbSiTi and AlCr targets in a mixed argon and nitrogen atmosphere. By adjusting the residence time of the substrates in the plasma regions of the AlCrNbSiTi and AlCr targets, multilayered thin films with varied bilayer periodic thicknesses ranging from 6 to 40 nm were fabricated. For the multilayer thin film with 15 nm bilayer period thickness, the thickness ratios of AlCrNbSiTiN and AlCrN single layer

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were adjusted to evaluate their influence on the hardness and corrosion resistance of films. XRD analysis indicated that all AlCrNbSiTiN/AlCrN multilayer films, as well as single-layer AlCrNbSiTiN and AlCrN films, exhibited a face-centered cubic crystal structure. Notably, the AlCrNbSiTiN/AlCrN multilayer film with a 15 nm bilayer period demonstrated a high hardness of 28 GPa and excellent corrosion resistance in 0.5 M H<sub>2</sub>SO<sub>4</sub> aqueous solution, with a corrosion impedance value of  $1.19 \times 10^6 \text{ W}\cdot\text{cm}^2$ . The influence of AlCrNbSiTiN to AlCrN thickness ratios on the mechanical properties and corrosion resistance of AlCrNbSiTiN/AlCrN multilayer thin film with 15 nm bilayer period was explored in this work.

9:20am **MA3-3-WeM-5 Impact of Microstructural Characteristics of HVOF-Deposited Cr<sub>3</sub>C<sub>2</sub>-Cermets on Their Performance in Sliding Abrasive Wear**, *Xinqing Ma, Peter Ruggiero*, Curtiss-Wright Corporate, USA  
Nowadays, Cr<sub>3</sub>C<sub>2</sub>-based cermet hardface coatings manufactured by advanced HVOF processes are well recognized for their corrosion and erosion resistance, particularly at high temperatures. Their lightweight nature and high temperature capability make them an attractive alternative to WC-based alloy coatings and hard Cr plating coatings. The objective of this study is to develop optimal Cr<sub>3</sub>C<sub>2</sub>-NiCr coatings by comparing different feedstock materials, including feedstock with nanocrystalline and/or submicron sized Cr<sub>3</sub>C<sub>2</sub> phases. The focus of the investigation is on understanding the impact of feedstock features such as particle size, morphology, and nanocrystalline carbide sizes, as well as sliding abrasive wear conditions on the coating properties and sliding wear performance. The results of the study indicate that the sliding wear resistance of the Cr<sub>3</sub>C<sub>2</sub>-NiCr coatings is highly influenced by the features of the Cr<sub>3</sub>C<sub>2</sub> carbides. With the special interest of nano-crystalline and/or submicron-sized carbides, the presence of nano, submicron and a few microns sized carbides in the coatings was revealed to improve their density, residual stress and hardness, leading to a significant reduction in wear rates under test conditions. Furthermore, the size of the abrasive SiC grit on the counter surface plays a significant role in determining the sliding wear behavior of these coatings. Based on the analysis of the test data, the mechanisms behind the performance of the Cr<sub>3</sub>C<sub>2</sub>-NiCr coatings have been investigated and used to interpret their sliding wear behaviors. This study has identified and recommended optimized materials for improved coating properties based on the key findings and results analyses. These findings and model analyses contribute to the understanding of the relationship between feedstock features, sliding abrasive wear conditions, and the wear rates of HVOF-sprayed Cr<sub>3</sub>C<sub>2</sub>-NiCr coatings. Hence, the optimized manufacture method by advanced HVOF method will meet the on-going need for a robust alternative solution to hard chromium plating (HCP) method.

9:40am **MA3-3-WeM-6 Interface Amorphization Controls Maximum Wear Resistance of Multi-Nanolayer Carbon/WC Coatings**, *Narguess Nemati*, Langtoftevej 9, Viby, Denmark

Multilayer coatings offer significant advantages in protecting materials' surfaces by shielding the underlying materials hierarchically from damage and wear. The layering morphology and structure of multilayer coatings directly affect their wear resistance capacity. Using a systematic set of experiments and molecular dynamic simulations, we studied the effect of layering thickness on the macroscale wear response of DLC/WC multi-nanolayer coatings. Our study revealed the existence of a critical bilayer thickness where maximum scratch hardness and wear resistance can be achieved. Our large-scale molecular dynamics simulations showed that reducing the WC layer thickness to a certain limit increases the scratch hardness due to the confinement of dislocation motion. However, when the thickness of WC layers falls below 2 nm, the deformation mechanism transitions from the interface-induced dislocation confinement to the interface-mediated amorphization of WC layers, reducing the scratch hardness of the coating. This finding offers a procedure for optimizing the macroscale wear performance of multi-nanolayer coatings.

11:00am **MA3-3-WeM-10 Improving the Elemental Accuracy and Imaging Precision in Atom Probe Tomography of TiSiN Coatings Using Isotopic Substitution and Peak Decomposition**, *Saeideh Naghdali, Maximilian Schiester*, Montanuniversität Leoben, Austria; *Marcus Hans*, RWTH Aachen University, Germany; *Markus Pohler, Christoph Czettl*, CERATIZIT Austria GmbH, Austria; *Michael Tkadletz, Nina Schalk*, Montanuniversität Leoben, Austria

Owing to its excellent mechanical properties TiSiN is commonly used as hard protective coating in cutting applications. However, the detailed investigation of the microstructure of TiSiN is a challenging task due to its nanocomposite structure, typically consisting of nanocrystalline and

amorphous regions. Atom probe tomography would be a valuable method to study the local elemental distribution with high resolution, but peak overlaps of Si and N in the mass spectrum do not allow for an unambiguous differentiation, resulting in poor elemental accuracy and imaging precision. In order to improve both, isotopic substitution of naturally abundant nitrogen with <sup>15</sup>N enriched nitrogen was applied, allowing to disentangle the contribution of Si and N to the mass spectrum. In addition, the bulk composition of TiSiN coatings deposited with naturally abundant nitrogen was corrected by peak decomposition considering the corresponding isotopic abundancies, resulting in an improved elemental accuracy. A spatially resolved approach via voxeling the 3D reconstructed data and subsequent peak decomposition of the individual voxels also allows the improvement of the imaging precision. The results showed, that Si is to some extent incorporated into a Ti<sub>1-x</sub>Si<sub>x</sub>N solid solution, but also Ti is incorporated into the amorphous Si<sub>x</sub>Ti<sub>1-x</sub>N<sub>2</sub> phase fraction.

11:20am **MA3-3-WeM-11 Comparison of the Protective Performance of YSZ Coatings on Austenitic Steel Under Static and Dynamic Molten Carbonate Conditions**, *M. Teresa de Miguel, Gustavo García Martín, M. Isabel Lasanta, Jaime Chaves, Francisco Javier Perez Trujillo*, Universidad Complutense de Madrid, Spain; *Pauline Audigé, Sergio Rodríguez, Alina Agüero*, Instituto Nacional de Técnica Aeroespacial (INTA), Spain

The development of protective coatings is crucial for mitigating the severe corrosion caused by molten salts operating at high temperatures in Concentrating Solar Power plants. While nitrate-based salts are currently used as thermal storage media in CSP plants, carbonate salts offer superior thermal stability. The properties of the Li-Na-K carbonate eutectic were enhanced by the addition of 0.5% of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> nanoparticles. The new mixture presents a reduced melting point and an increase in the decomposition temperature, allowing the operation of the CSP plants at 700 °C. This increase would enhance steam generation efficiency and would reduce the Levelized Cost of Electricity (LCoE). However, the intense corrosive effects of molten carbonates demand the development of durable protective coatings to extend the lifespan of critical components.

This study evaluates the performance of yttria-stabilized zirconia (YSZ) sol-gel coatings applied to the 310H austenitic steel when it is exposed to static and dynamic conditions in a lab-scale setup. Corrosion tests were conducted at 700°C for up to 2500 hours in the eutectic ternary Li<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>CO<sub>3</sub>-K<sub>2</sub>CO<sub>3</sub> mixture with  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> additive. The protective behavior of the coatings was assessed through gravimetric analysis, microstructural characterization and XRD. The performance of the coated material was also compared with the uncoated substrate. The coated samples exhibited an improved corrosion resistance, whereas the uncoated steels showed substantial degradation, including detachment and high mass variation. The presence of the YSZ coating reduced the corrosion extent by over one-third, with the thickness of the corrosion products measuring approximately 200  $\mu\text{m}$  - 300  $\mu\text{m}$  on the uncoated substrate, and between 50  $\mu\text{m}$  - 70  $\mu\text{m}$  on the coated samples.

The YSZ coating exhibited very similar behavior under both dynamic and static conditions. No significant difference was observed in the thickness of the corrosion product layer. Additionally, the same multilayer structure was identified, with the outer layer mainly composed of LiFeO<sub>2</sub>, while the inner region was enriched in iron and chromium oxides. However, the uncoated austenitic steel showed higher degradation when exposed to dynamic conditions, displaying cracks along the corrosion layer and detachments.

These findings highlight the potential of YSZ coatings to enhance the durability of structural materials in next-generation CSP plants employing molten carbonates.

11:40am **MA3-3-WeM-12 Superhard Single-phase Ti<sub>1-x</sub>Al<sub>x</sub>B<sub>y</sub> Films with Good Oxidation Resistance Grown without External Heating using Hybrid HiPIMS/DCMS Technique**, *Bartosz Wicher*, Linköping University, IFM, Thin Film Physics Division, Poland; *Vladyslav Rogoz*, Linköping University, IFM, Thin Film Physics Division, Ukraine; *Oleksandr Pshyk*, Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland, Ukraine; *Szilard Kolozsvari, Peter Polcik*, Plansee Composite Materials GmbH, Germany; *Ivan Petrov*, University of Illinois at Urbana-Champaign, USA, Bulgaria; *Lars Hultman, Grzegorz Greczynski*, Linköping University, IFM, Thin Film Physics Division, Sweden

A hybrid High-Power Impulse Magnetron Sputtering (HiPIMS) and Direct Current Magnetron Sputtering (DCMS) approach with TiB<sub>2</sub> and AlB<sub>2</sub> targets is used to grow Ti<sub>1-x</sub>Al<sub>x</sub>B<sub>y</sub> thin films with  $0.40 \leq x \leq 0.76$  and  $1.81 \leq y \leq 2.03$ . The hybrid sputtering method ensures precise control over the energy and momentum of ionized species. The primary aim is to optimize the Al content for enhancing the high-temperature oxidation resistance while

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maintaining excellent mechanical properties that stem from the diboride structure. No external substrate heating is used resulting in the substrate temperature lower than 180°C.

Oxidation tests performed at temperatures ranging from 700 to 900 °C indicate a substantial improvement in oxidation resistance with higher Al content. Films with  $x \leq 0.49$  develop porous, B-depleted oxide layers exhibiting titanium dioxide (TiO<sub>2</sub>) rutile phase structure and often show spallation. In contrast, the Ti<sub>1-x</sub>Al<sub>x</sub>B<sub>y</sub> layers with  $x \geq 0.58$  form dense oxide scales composed of amorphous alumina (Al<sub>2</sub>O<sub>3</sub>) and borate (Al<sub>18</sub>B<sub>4</sub>O<sub>33</sub>) phases, which effectively passivate the film surface against further oxidation. The oxide scales formed on the high-Al content films exhibit nanoindentation hardness comparable to that of TiAlN coatings and very good adhesion to the underlying substrate due to better matching of thermal expansion coefficients.

These findings offer a promising foundation for developing high-performance boride-based coatings for applications in industries such as aerospace and power generation that require coating materials with mechanical strength and resistance to high-temperature oxidation.

## Acknowledgment

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12:00pm **MA3-3-WeM-13 Sputter Deposition of Ultrathick Boron Carbide Coatings on Rolling Spherical Substrates**, *William Rios Lopez, James Merlo, Greg Taylor, Jean-Baptiste Forien, Sergei Kucheyev*, Lawrence Livermore National Lab, USA

Advancing inertial confinement fusion (ICF) technology requires the development of novel vapor deposition processes for the fabrication of spherical ablators. Boron carbide (B<sub>4</sub>C) is a promising material for next-generation ICF fuel ablator capsules due to its unique properties, enabling optimal ICF implosion dynamics. Furthermore, its compatibility with direct-current magnetron sputtering (DCMS) offers scalability for ablator production. However, the deposition of B<sub>4</sub>C films on spherical substrates remains challenging due to the complex interplay between directional deposition processes and substrate geometry. This study focuses on minimizing the density of nodular defects in ultrathick B<sub>4</sub>C coatings deposited by DCMS on rolling spherical substrates. We used a custom-designed substrate holder to systematically study how the rolling kinetics of spherical substrates influences the nodular defect population in coatings. This work was performed under the auspices of the U.S. DOE by LLNL under Contract DE-AC52-07NA27344.

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