Protective and High-temperature Coatings
Room Palm 3-4 - Session MA1-1-MoM

Coatings for High Temperatures and Harsh Environment Applications I

Moderator: Francisco Javier Perez Trujillo, Universidad Complutense de Madrid, Spain

10:00am **MA1-1-MoM-1 Improving the Lifetime and Efficiency of Next Gen Aircraft Turbine Engines with PVD***, **Thibault Maerten [thibault.maerten@oerlikon.com]**,* Oerlikon Balzers Coating AG, France

Aircraft engine operating conditions continue to push the limits of material capabilities, which requires robust, multifunctional, and durable coating solutions for turbine hot-sectioncomponents. These turbines are mainly made from metallic components that are protected by thermal barrier coatings (TBCs). A TBC system is usually formed by a top ceramic layer deposited onto a bond layer over the substrate. MCrAlY (where M = Ni, Co, Fe or combinations of these metals) coatings are widely used as bond-coat material on blades and vanes due to their good resistance against oxidation and corrosion at high temperature. MCrAlY coatings are typically applied through thermal spray, electroplating and CVD methods. However, with ever-increasing turbine combustion temperatures, rotation speed for the sake of increased efficiency, the classic deposition methods are approaching their functional limits.

Significant developments over the last decade have made Physical Vapor Deposition (PVD) technologies increasingly compelling for MCrAlY coating deposition. PVD MCrAlY coatings exhibit high deposition rate, excellent surface adhesion, high density, and exceptional uniformity on complex geometries such as airfoils. Additionally, PVD MCrAlY coatings show a significantly lower surface roughness (Ra = 3-4 µm), which can be further smoothed by post-treatment, benefiting the application of EB-PVD TBCs.

With these significant advantages, PVD MCrAlY coatings produced by cathodic arc offer new opportunities for next gen aircraft engines development. In this talk, we aim to present several case studies involving replacement by PVD of traditional methods to produce MCrAlY. The coating properties (microstructure, composition, thickness distribution) and resulted performances (fatigue debit, oxidation and corrosion resistance) will be presented and compared.

10:20am **MA1-1-MoM-2 Mechanisms of Solid Particle Erosion in Aerospace Materials and Protective Coatings***, **Stephen Brown [stephen.brown@polymtl.ca]**[1], Etienne Bousser, Benjamin Milan-Ramos,* Polytechnique Montréal, Canada*; Juan Manuel Mendez,* MDS Coating Technologies, Canada*; Marjorie Cavarroc-Weimer,* Safran Tech, France*; Ludvik Martinu, Jolanta Ewa Klemberg-Sapieha,* Polytechnique Montréal, Canada

Solid particle erosion (SPE) is a tribological process involving material removal by repeated impacts of high-velocity particles. Despite years of research, fundamental mechanisms governing SPE remain poorly understood, particularly those concerning the erosion of metals at 90° impingement and the deformation of protective coatings in the elasto-plastic erosion regime. This work presents a detailed erosion study of bare Ti-6Al-4V and protective TiAlN-based coatings under varied particle velocity (50-120 m/s), impingement angle (15°-90°), and particle type/size (Al_2O_3, crushed glass 50-140 µm). Beyond standard metrics such as scar depth and volume loss rates, the eroded surfaces were extensively characterized by Plasma Focused Ion Beam cross-sectioning (PFIB), Electron Backscatter Diffraction (EBSD), Transmission Electron Microscopy (TEM) of eroded lamellae with Selected Area Diffraction, Transmission Kikuchi Diffraction (TKD), and nanoindentation mapping.

For Ti-6Al-4V, full erosion tests were compared to single particle impacts; both approaches showed cloudy microstructures indicative of severe local strain near impact sites, confirmed by TEM to be nanocrystalline. The affected depth did not exceed 7 µm, and nanoindentation revealed an 11% hardness increase. Extensive particle embedment occurred during multi-impact tests, yet damage morphology and affected depth were near-identical to single impacts, challenging cumulative wear models and suggesting that the 90° erosion of Ti-6Al-4V can be represented as the sum of individual impacts.

TiAlN coatings deposited via cylindrical magnetron sputtering exhibited architecture-dependent failure. Monolithic TiAlN initially degraded by nano-chipping, along with plastic deformation of the columnar structures 100-200 nm into the subsurface. This was followed by catastrophic adhesive failure after the coating thins beyond a certain threshold. A multilayer TiAlN/TiAl system exhibit a similar but distinct failure mode: cracks propagated along TiAl interlayers, promoting local delamination of the overlying TiAlN. In essence the same threshold-type failure occurs, however, the TiAl interlayers decrease the TiAlN layer thickness and thus the distance to the nearest interface. The result is progressive layer-by-layer material removal rather than bulk spallation, offering insight into how architecture governs erosion resistance.

10:40am **MA1-1-MoM-3 Microstructure and Oxidation of PVD Coatings on TiAl and Ni Superalloys for High-Temperature Applications***, **Radosław Swadźba [radoslaw.swadzba@git.lukasiewicz.gov.pl]**,* Łukasiewicz Research Network - Uppersilesian Institute of Technology, Poland **INVITED**

Modern aircraft engines operate at increasingly higher temperatures to improve thermal efficiency and reduce fuel consumption. These extreme conditions place severe oxidation and corrosion demands on structural materials such as TiAl intermetallics and Ni-based superalloys. Although these alloys combine excellent strength-to-weight ratios with good high-temperature mechanical properties, their long-term performance depends strongly on effective surface protection. The development of advanced oxidation-resistant coatings is therefore essential for enabling higher operating temperatures and extending the lifetime of next-generation aeroengine components.

This talk presents recent work on the development, microstructural design, and oxidation behaviour of protective coatings produced by the Closed Hollow Cathode Physical Vapor Deposition (CHC-PVD) method on TiAl and Ni-based substrates. The CHC-PVD process offers high plasma ionization, allowing deposition of thick, adherent, and compositionally complex coatings with tailored architectures.

The coating systems investigated include Ti–Al–Cr alloys modified with Si and Y, MAX phase coatings (Ti_2AlC and Cr_2AlC) on γ-TiAl, and MCrAl-type coatings on Ni-based superalloys. Detailed characterization was performed using High-Resolution Transmission Electron Microscopy (HRTEM), Scanning Transmission Electron Microscopy (STEM), and high-temperature X-ray diffraction (HT-XRD) to study both as-deposited coatings and their phase evolution during heat treatment. These advanced techniques made it possible to reveal nanolaminate microstructures, analyze interfaces, and examine thermally grown oxides in detail.

High-temperature oxidation studies under isothermal and cyclic conditions revealed clear differences in performance among the investigated coatings. The degradation modes, along with the formation and evolution of protective alumina scales, were examined in detail using HRTEM and STEM to establish correlations between microstructure, composition, and oxidation behavior.

The results highlight the potential of the CHC-PVD technique for producing advanced high-temperature coatings with optimized microstructures and oxidation behavior, contributing to the development of durable, lightweight materials for future aircraft engines.

11:20am **MA1-1-MoM-5 Predictive Analytics of Aluminide Diffusion Coatings Using Machine Learning to Forecast Their Aging and Service Life***, **Vladislav Kolarik [vladislav.kolarik@ict.fraunhofer.de]**, Maria del Mar Juez Lorenzo,* Fraunhofer Institute for Chemical Technology ICT, Germany*; Pavel Praks, Renata Praksová,* IT4Innovations National Supercomputing Center, VSB - Technical University of Ostrava, Czechia

Aluminide diffusion coatings offer a reliable and economical way to protect steel from high-temperature corrosion in harsh environments. These coatings can be applied as aluminum slurries using various deposition methods, including spraying or brushing, and are subsequently heat-treated to form the diffusion layer. Predictive analytics using machine learning offers great potential to forecast the aging behavior and lifetime of the coating under operating conditions. Machine learning predictions rely solely on historic data and do not require physical models to describe dependencies. This is especially advantageous for systems influenced by multiple parameters, as machine learning can identify patterns and relationships that humans cannot. Regression-based predictive models, such as Symbolic Regression or decision-tree algorithms like CatBoost and XGBoost, have proven to be suitable.

Two key variables were identified for describing the service life of an aluminum diffusion coating: (1) the ratio of the inner Fe_3Al layer to the total

[1] **Graduate Student Award Finalist**

coating thickness, and (2) the aluminum concentration in the Fe_3Al layer. The first variable indicates a milestone in the coating's service life, occurring when the ratio equals 1. At this point the diffusion coating evolves into a single aluminum-poor layer. The aluminum concentration in this single layer reflects the amount of aluminum remaining in the coating, which is essential for forming a protective alumina layer. The input parameters, time, temperature, atmosphere, overall coating thickness, thicknesses of the partial layers, number of the partial layers, type of slurry etc., were collected from our previous research as well as from literature. The transition to a single-layer coating was forecasted to occur after 28,000 hours at 650°C in air, following a time law close to parabolic, indicating that diffusion dominates the process. The aluminum content remains in the range of 25 at% over 100,000 hours, indicating that Fe_3Al will still be present.

The research shows that machine learning is very effective in analyzing complex material systems affected by multiple parameters, where understanding the relationships and importance of these parameters is difficult using conventional physical modeling approaches.

11:40am **MA1-1-MoM-6 Tailored Formation of Intermetallic Phases in Nanolayered Metallic Systems***, **Vincent Ott [vincent.ott@kit.edu]**, Sven Ulrich, Michael Stüber,* Karlsruhe Institute of Technology (KIT), Institute for Applied Materials (IAM), Germany

The temperature induced formation and stability of phases in nanoscale multilayer thin films are strongly affected by confinement effects and interface-controlled kinetics. As the characteristic dimensions of the layers are reduced, the thermodynamic driving forces and atomic mobility change, leading to distinct size-dependent phase formation behavior during heat treatment. In metallic multilayers, this often results in a modified or entirely suppressed sequence of equilibrium phase transformations. Using the Ru/Al model system and its ternary extensions with Hf, Cr, and Cu, we demonstrate that decreasing the individual layer thicknesses can kinetically inhibit the formation of equilibrium phases while promoting the preferential stabilization of the crystallographically simple cubic B2 structure. This enables the controlled synthesis of metastable intermetallic alloys beyond the thermodynamic equilibrium regime. The phase evolution was monitored in-situ by high-temperature X-ray diffraction (HT-XRD), while complementary electron microscopy and atom probe tomography (APT) provided insight into the resulting nanoscale structure and chemical distribution. The general validity of this kinetic stabilization concept is further illustrated by the Fe–Ti system, in which the B2 FeTi phase can be selectively formed at comparable nanometer-scale periodicities. These findings highlight the potential of nanoscale layering to engineer novel metastable phases with tailored structural and functional properties.

Protective and High-temperature Coatings
Room Palm 3-4 - Session MA1-2-MoA

Coatings for High Temperatures and Harsh Environment Applications II

Moderators: Vladislav Kolarik, Fraunhofer Institute for Chemical Technology ICT, Germany, **Fernando Pedraza**, La Rochelle University, Laboratory LaSIE, France

1:40pm **MA1-2-MoA-1 Synergistic Effects of Ta and Si Alloying on the Longterm Oxidation and Hot Corrosion Resistance of Ti–Al–N Coatings***,* ***Anna Hirle [anna.hirle@tuwien.ac.at]****, Rainer Hahn, Oliver E. Hudak, Philip Kutrowatz, Tomasz Wojcik,* Christian Doppler Laboratory for Surface Engineering of High-performance Components, TU Wien, Vienna, Austria*; Szilard Kolozsvári, Peter Polcik,* Plansee Composite Materials GmbH, Lechbruck am See, Germany*; Anders.O Eriksson, Carmen Jerg, Klaus Boebel,* Oerlikon Balzers, Oerlikon Surface Solutions AG, Balzers, Liechtenstein*; Helmut Riedl,* Christian Doppler Laboratory for Surface Engineering of High-performance Components, TU Wien, Vienna, Austria; Institute of Materials Science and Technology, TU Wien, Vienna, Austria

$Ti_{1-x}Al_xN$ is one of the most used coating materials applied in various applications, including i.e. machining and forming tools but also components, due to its excellent thermomechanical properties. However, as operating temperatures rise, new strategies are needed. Alloying $Ti_{1-x}Al_xN$ with Ta or Si shows promise. In more detail, the incorporation of Ta shifts the onset of the spinodal decomposition towards higher temperatures. Furthermore, the formation of the unfavoured anatase phase during oxidation is suppressed and the rutile phase is stabilised [1,2]. Adding Si enhances the thermal stability and oxidation resistance, while concomitantly leading to the formation of a nanocomposite microstructure [3]. Recent research explores combined alloying with Ta and Si to improve both oxidation resistance and mechanical performance. [4,5]. Compared with $Ti_{1-x}Al_xN$ coatings – which fully oxidize at 1000 °C (15 h, synthetic air) – $Ti_{1-x-y-z}Al_xTa_ySi_zN$ thin films form oxide scales below 1 µm [5].

Furthermore, $Ti_{1-x-y-z}Al_xTa_ySi_zN$ coatings exhibit excellent mechanical properties, making them promising candidates for high temperature applications [4].

The present study investigates a series of $Ti_{1-x-y-z}Al_xTa_ySi_zN$ coatings deposited by cathodic arc evaporation using an industrial-scale Oerlikon Balzers INNOVA 1.0 system. Two distinct target compositions were utilised, along with varying deposition parameters. Long-term oxidation experiments were conducted in a conventional furnace at temperatures of 850 °C for durations of 24 h up to 500 h. After the oxidation processes, we conducted an analysis using X-ray diffraction (XRD), focused ion beam (FIB) techniques, and transmission electron microscopy (TEM). Additionally, we performed low-temperature hot corrosion (LTHC) experiments at 700 °C with a hot gas corrosion testing rig, varying the concentrations of SO_2.

In summary, the study demonstrates that $Ti_{1-x-y-z}Al_xTa_ySi_zN$ coatings exhibit extremely low oxidation kinetics, suggesting long-term stability well beyond 500 hours, alongside excellent resistance to hot gas corrosion environments. Based on these results, cathodic arc-evaporated Ti-Al-N-based coating materials are also promising candidates for protective applications in the aviation and power generation sectors.

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[3] Z.R. Liu et al., J. Alloys Compd. 917 (2022) 165483.
[4] A.R. Shugurov et al., Vacuum. 216 (2023) 112422.
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2:00pm **MA1-2-MoA-2 Fabrication, Characterisation and Tribological Testing of Magnetron Sputtered Cr Coated Zr Alloy Cladding for Enhanced Accident Tolerance in Light Water Reactors***, Thais Netto,* Manchester Metropolitan University, Brazil*; Adele Evans,* Manchester Metropolitan University, UK*; David Goddard, Jack Cooper,* United Kingdom National Nuclear Laboratory, UK*;* ***Peter Kelly [peter.kelly@mmu.ac.uk]****,* Manchester Metropolitan University, UK

Research into accident-tolerant fuels (ATFs) for light water reactors (LWRs) has focused on improving the safety of zirconium alloy fuel rod claddings and one of the more developed approaches is the use of chromium coatings deposited onto the claddings. In addition to performing in oxidising conditions, normal operation also causes fretting wear on the fuel rod surface, which requires tribological improvements.

The aim of this work, therefore, is to produce Cr coatings using the magnetron sputtering technique for Zr alloy nuclear fuel rod cladding material to enhance oxidation and mechanical resistance. The coatings were characterised, as a function of deposition conditions, in terms of their morphology, topography, hardness, reciprocating and fretting wear resistance, scratch test performance and oxidation resistance in autoclave and air oxidation tests.All the coatings provided excellent oxidation protection, in comparison to the uncoated samples. Mechanical testing indicated contrasting results with coatings with higher hardness showing enhanced wear protection, but lower coating hardnesses provided better scratch test performance and reduced fretting wear.

Scale up of these experiments has progressed from small flat coupons, through short (<20cm) rods, to full length (4m) fuel rods.

2:20pm **MA1-2-MoA-3 Second Phase-Driven Surface Engineering Strategies for Corrosion and Oxidation Protection of Mg–8Al–4Ca Alloy***,* ***Yueh-Lien Lee [yuehlien@ntu.edu.tw]****,* National Taiwan University, Taiwan **INVITED**

This study elucidates how second phases, particularly the β-Al-Ca intermetallic, influence the behavior and corrosion performance of the non-flammable Mg–8Al–4Ca alloy during cerium conversion coating and micro-arc oxidation (MAO) treatments. Scanning Kelvin probe force microscopy (SKPFM) and transmission electron microscopy (TEM) analyses reveal that the β phase exhibits a lower Volta potential and higher electrochemical activity than the α-Mg matrix, serving as a micro-galvanic anode that accelerates localized corrosion and hydrogen evolution. During the cerium conversion process, this activity disrupts film uniformity; however, a simple deionized-water pretreatment dissolves the exposed β phase into $Al(OH)_3$, promoting homogeneous Ce deposition with enhanced coating integrity and corrosion resistance. In MAO processing, the distribution and conductivity of β phases strongly affect discharge behavior and coating development. At low voltages, localized discharges near β regions lead to thinner and non-uniform films, while higher voltages facilitate the formation of Mg–Ca-rich silicate/oxide phases that improve corrosion resistance but hinder further thickening. Selective removal of surface β phases prior to MAO yields thicker and more uniform coatings. These findings clarify the mechanistic link between second-phase characteristics and coating evolution, providing effective strategies to engineer durable protection for non-flammable magnesium alloys.

3:00pm **MA1-2-MoA-5 Development of High-Temperature Ceramic Bond Coats for Environmental Barrier Coatings***,* ***Rebekah Webster [rebekah.webster@nasa.gov]****, Benjamin Kowalski, Bryan Harder,* NASA Glenn Research Center, USA

Environmental barrier coatings (EBCs) have enabled the use of silicon carbide (SiC)-based ceramic matrix composites (CMCs) in gas turbine engines by protecting the underlying CMC from corrosive combustion species such as water vapor. EBCs currently in service consist of a silicon bond coat and a rare earth silicate topcoat. The melting point of the silicon bond coat (~1410°C) limits the upper use temperature of these coating systems. To protect SiC-based CMCs at temperatures beyond that achievable by the current state-of-the-art, a mullite-based bond coat capable of withstanding temperatures of up to 1482°C has been developed at NASA Glenn Research Center. The mullite-based bond coat can be deposited by either plasma spray physical vapor deposition (PS-PVD) or slurry processing, and various EBC architectures with this bond coat have been developed and tested. In this work, the performance of these EBCs under oxidizing environments including steam and temperature cycling are reviewed, with the effect of coating microstructure, namely porosity, on oxidation being highlighted.

3:20pm **MA1-2-MoA-6 Statistical Correlation between Microstructural Features and Process Forces in Conventional and Ultrasonic-Assisted Milling of Plasma Claddings***, Kai Treutler,* TU Clausthal, Germany*;* ***Dirk Schröpfer [dirk.schroepfer@bam.de]****,* Bundesanstalt für Materialforschung und -prüfung, Germany*; Maraike Willeke,* TU Clausthal, Germany*; Thomas Kannengießer,* Bundesanstalt für Materialforschung und -prüfung, Germany*; Volker Wesling,* TU Clausthal, Germany

The development of highly efficient and economical steel components in plant and process engineering is crucial for reducing CO2 emissions. To withstand the high combined corrosive, tribological, thermal, and mechanical stresses, wear-resistant coatings tailored to the application and steel grade are employed. The increasing demand to substitute conventional cobalt alloys with nickel alloys, coupled with the need for defined or functional surfaces of high integrity, necessitates the development of novel wear-resistant coatings.

The use of wear-resistant coatings is essential for highly efficient and economical steel components in equipment, process, and power plant engineering. Co-alloys are commonly used as wear-resistant coatings for steel components, tailored to the specific application. The substitutability of Co alloys with Ni-based wear protection systems, in addition to price and supply uncertainties, is facilitated by the combination of innovative welding and machining processes such as ultrasonic-assisted milling.

This study investigates the influence of the microstructure and precipitation morphology adjusted by means of alloy modification on the machinability of wear-resistant plasma cladded coatings. The wear protection alloy NiCrMoSiFeB (trade name: Colmonoy 56 PTA), typically used for screw machines, is employed as a model system. Metallurgical investigations and in-situ measurements of occurring process forces and temperatures at the tool cutting edge during milling, as well as subsequent investigations of tool wear and surface integrity, allow for a detailed analysis and correlation between microstructural properties and machinability.

The primary objective of this study is the statistical correlation between specific microstructural features, like precipitation size, shape and amount with the characteristic process forces of conventional and ultrasonic assisted milling of the claddings.

The addition of Al, Ti, or Nb to the cast samples results in a clear change in the microstructure, hardness and machinability. Al and Ti cause long-needled or star-shaped precipitations and hardness increases, which lead to higher cutting forces and increased tool wear. In the case of the modified alloys, the inclusion of the alloying element Nb results in the formation of a more refined hard phase and reduces the machining force required for C56.

In most cases, the wear resistance potential has been maintained. The statistical model allows to adjust the chemical composition to a better machinability of the hard facings.

4:00pm **MA1-2-MoA-8 Oxidation Resistance of Binary and Ternary Nitrides Obtained by Magnetron Sputtering**, ***Ludovic Mereaux [ludovic.mereaux@unilim.fr]***, IRCER, France; *Edern Menou, Thomas Vaubois,* Safran, France; *Cédric Jaoul,* IRCER, France; *Marjorie Cavarroc,* Safran, France

Increasing aircraft engine temperature is one method, amongst others, to decarbonize aviation. But at high temperature, metallic materials performances are drastically decreased due to the effect of hot corrosion. To limit this impact, metallic materials need to be protected with dedicated coatings with adequate properties, which “entropy-augmented” ceramics could feature.

However, the composition space of complex ceramics is very wide, and comparatively very few bibliographical data are available as these specific ceramics have not been widely studied to date. While the use of data-driven approaches to identify relevant compositions appears necessary, it is not sufficient as (1) it requires data to be trained on, and (2) final properties should be experimentally assessed.

Nitride coatings obtained by PVD methods have been used in the machine tool and aerospace industries for many years. Binary nitride systems exhibit mechanical properties such as high hardness (20-25 GPa [1]). The addition of transition metals, such as Al or Si, improves physical and chemical properties like wear resistance, thermal stability and oxidation resistance [2].

Two main challenges have to be overcome: achieving a single solid solution film to guarantee both material and property homogeneity throughout the coatings, and assessing the long-term mechanical and environmental stability of the materials.

It was decided to creates our own database, starting from simple binary coating with the progressive addition of elements. In this talk, we will present results on the oxidation of binary and ternary nitride coatings. These coatings are obtained by magnetron sputtering in a reactive atmosphere and they are annealed in air up to 900°C, to propose oxidation mechanisms.

[1] W. D. Sproul, M. E. Graham, M.-S. Wong, et P. J. Rudnik, « Reactive unbalanced magnetron sputtering of the nitrides of Ti, Zr, Hf, Cr, Mo, Ti-Al, Ti-Zr and Ti-Al-V », *Surface and Coatings Technology*, vol. 61, nº 1, p. 139–143, déc. 1993, doi: 10.1016/0257-8972(93)90216-B.

[2] V. Novikov, N. Stepanov, S. Zherebtsov, et G. Salishchev, « Structure and Properties of High-Entropy Nitride Coatings », *Metals*, vol. 12, nº 5, p. 847, mai 2022, doi: 10.3390/met12050847.

4:20pm **MA1-2-MoA-9 Adaptive Opto-Neuromorphic Device Based on Monolayer MoS_2 for Extreme-Temperature Cognitive Operations**, ***Pukhraj Prajapat [pukhraj.npl@gmail.com]***, *Govind Gupta,* National Physical Laboratory, India

High-temperature neuromorphic devices are becoming increasingly essential as technology advances to support space exploration and withstand extreme conditions, such as those found in factories. To overcome this need, the researchers are devising technologies that imitate the human brain structure and operation. In this work, we present a scalable neuromorphic device based on a monolayer of MoS_2 that demonstrates operation at 100°C. The device portrays excellent electrical performances, mostly due to the great thermal stability of monolayer MoS_2 and its mechanical flexibility. Among these performances are low power consumption, fast switching, high resistance ratio, low switching voltage, and long stable endurance (~10^3 cycles). Besides, the device mimics neuromorphic behaviour by embedding the synaptic plasticity that is the major functional property of biological neural networks, thus allowing advanced cognitive computing in extreme environments. This is the first step toward a combination of materials science and neuromorphic computing, and it clears the way for smart resilient electronics that could survive in a variety of harsh conditions. This research aimsto achieve a significant breakthrough in the field of high-temperature electronics, paving the way for the development of future high-performance electronics that can meet the demands of modern technology.Keywords: 2D, TMDCs, Neuromorphic, Brain inspired, MoS2

4:40pm **MA1-2-MoA-10 Reactive Sputtering of CrMoNbWxTiCy Carbide Films by High Power Impulse Magnetron Sputtering System: Effect of W and Carbon Contents**, ***ChunHao Cheng [itsjonardgx@gmail.com]***, *Yung-Chin Yang,* National Taipei University of Technology, Taiwan; *Jyh-Wei Lee,* Ming Chi University of Technology, Taiwan; *Bih-Show Lou,* Chang Gung University, Taiwan; *Chia-Lin Li,* Ming Chi University of Technology, Taiwan

High power impulse magnetron sputtering (HiPIMS) has attracted significant attention for its ability to generate high-density plasma and achieve highly ionized metal species. Compared with conventional DC sputtering, this technique enhances ion bombardment energy, leading to improved film densification and adhesion. In this study, CrMoNbTiWx and CrMoNbWxTiCy high entropy alloy (HEA) coatings were deposited using HiPIMS to investigate the effects of tungsten and carbon incorporation on the microstructure and multifunctional properties of HEA carbide films. By varying the W target contents and controlling the reactive acetylene gas flow rates, the influence of target poisoning on film growth behavior was systematically analyzed. The film morphology and phase structure will be examined using field-emission scanning electron microscopy, X-ray diffraction, transmission electron microscopy, and atomic force microscopy. Mechanical properties, including hardness, adhesion, and wear resistance, will be characterized by nanoindentation, scratch, and pin-on-disk wear tests. Corrosion and oxidation resistance will be evaluated through a potentiodynamic polarization test in 3.5 wt.% NaCl solution and thermogravimetric analysis, while electrical performance will be assessed using a four-point probe to measure their electrical resistivities. This research aims to elucidate the roles of tungsten and carbon contents, as well as target poisoning behavior, in optimizing the structural, mechanical, and electrochemical performance of CrMoNbTiWxCy HEA carbide coatings, demonstrating the advantages of HiPIMS for developing dense and durable multifunctional protective films.

5:00pm **MA1-2-MoA-11 Materials for Aerospace Extreme Environments**, ***Samir M. Aouadi [samir.aouadi@unt.edu]***, University of North Texas, USA
INVITED

Protective and High-temperature Coatings
Room Town & Country C - Session MA4-1-MoA

Boron-containing Coatings I

Moderator: Martin Dahlqvist, Linköping University, Sweden

2:40pm **MA4-1-MoA-4 Charge Trapping Behavior in BN Films Fabricated by a Reactive Plasma-Assisted Coating Technique and Their Design Strategies**, ***Koji Eriguchi [eriguchi.koji.8e@kyoto-u.ac.jp]***, Kyoto University, Japan
INVITED

Boron nitride (BN) possesses highly desirable properties for a wide variety of industrial applications [1]. Its properties strongly depend on its microscopic structure: sp^2-bonded hexagonal (h-BN) and sp^3-bonded cubic (c-BN). For example, h-BN films are expected to be superior dielectric materials for electronic devices owing to their high dielectric breakdown field [2], whereas c-BN films have attracted considerable attention because of their high hardness [3]. Historically, using plasma-based technologies, these microscopic structures have been controlled predominantly by the energy of incident ions and the fluxes of B and other species [4]. However, crucial issues remain to be solved—namely, the degradation of dielectric breakdown lifetime for h-BN films and delamination due to residual stress in c-BN films. A fundamental understanding of the various degradation mechanisms in BN films is therefore required.

In this study, we formed BN films and their stacked structures with various bonding phases on Si substrates using a reactive plasma-assisted coating (RePAC) method [5]. After confirming their bonding networks by Fourier-transform infrared spectroscopy and optical properties by spectroscopic ellipsometry [6], we investigated the dielectric degradation of h-BN and the delamination behavior of c-BN in terms of charge trapping dynamics using Al/BN/Si devices. Characteristic charge trapping behaviors during time-dependent dielectric breakdown measurements were enhanced by the bombardment of higher-energy ions and the incorporation of impurities during h-BN film formation [7]. The presence of trapped charges was identified at the c-BN/Si interface and even within stacked BN films (h-/c-BN or c-/h-BN) [8]. In addition, film delamination was found to occur preferentially at the stacked c-/h-BN interface. Moreover, we found that time-dependent film delamination was closely correlated with the trapped charge density. These findings indicate that controlling charge trapping behaviors is key to improving the properties of BN films for various industrial applications.

This work was supported by JSPS KAKENHI (23H01728) and O.S.G. Foundation.

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[7] Y. Asamoto et al., J. Appl. Phys. **137**, 105301 (2025).

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3:20pm **MA4-1-MoA-6 Development of TiB_2:h-BN:a-C Based Nanocomposite Coatings with Enhanced Wear and Corrosion Resistance for Turbojet and Gas Turbine Components**, ***Gokhan Gulten [gokhangulten@atauni.edu.tr]***, *Banu Yaylali, Mustafa Yesilyurt, Ali Emre, Yasar Totik,* Atatürk University, Turkey; *Justyna Kulczyk-Malecka, Peter Kelly,* Manchester Metropolitan University, UK; *Ihsan Efeoglu,* Atatürk University, Turkey

Turbojet and gas turbine engines operate under severe thermo-mechanical and chemically aggressive conditions where simultaneous control of friction, wear, and corrosion is essential. This study reports the design and synthesis of a solid-lubricating nanocomposite architecture based on TiB_2:h-BN:a-C deposited by closed-field unbalanced magnetron sputtering (CFUBMS) driven by a hybrid HiPIMS + pulsed-DC power setup. The coating concept employs the synergistic combination of hard TiB_2 domains (load-bearing), hexagonal BN (lamellar solid lubricant, thermal stability), and amorphous carbon (low shear, transfer-film formation). To promote enhanced substrate adhesion and gradient stress accommodation on aerospace alloys (Inconel 718 and Ti-6Al-4V), a thin Cr adhesion layer and a CrN transition layer are incorporated. A Taguchi L9 experimental design is employed to map the influence of TiB_2 target voltage, N_2 flow, duty cycle, and working pressure on structure–property relationships. Comprehensive characterization includes XRD, Raman and XPS, SEM cross-sections, nanoindentation, scratch testing, and pin-on-disk tribometry at room and elevated temperatures. Electrochemical performance is assessed by potentiodynamic polarization and EIS to quantify corrosion resistance. The hybrid power delivery enhances ionization and adatom mobility, producing dense microstructures and superior adhesion. Process–structure–property correlations reveal reduced friction, improved wear resistance, and enhanced corrosion protection, establishing TiB_2:h-BN:a-C coatings as promising candidates for extending component life and reducing maintenance in advanced propulsion systems.

4:00pm **MA4-1-MoA-8 Energy Efficiency in Pulsed-DC Powder-Pack Boriding: A Sustainable Approach to Surface Hardening of Metallic Materials**, ***Ivan E Campos Silva [icampos@ipn.mx]***, Instituto Politecnico Nacional, Mexico **INVITED**

Boriding has emerged as an efficient thermochemical treatment to enhance the wear and corrosion resistance of metallic materials. The resulting boride layer, characterized by its exceptional hardness and outstanding thermal and chemical stability, outperforms nitrided, carburized, and PVD-coated surfaces. However, conventional powder-pack boriding still faces critical challenges (mainly the need for long treatment durations and high temperatures ((≥850 °C)) to achieve protective boride layers (50–75 µm thick). These conditions result in high energy consumption and increased production costs, limiting its industrial sustainability.

The pulsed-DC powder-pack boriding offers a sustainable alternative to conventional method by drastically reducing energy usage and processing time. This technique employs an electric field generated by a power source and a polarity-switching device connected to electrodes immersed in a powder mixture together with the metallic specimen. Remarkably, successful treatments have been performed at lower temperatures (600–750 °C) and shorter durations (up to 1.5 h), producing boride layers with excellent wear and friction performance—an unprecedented advancement in the field of solid boriding media and aligned with the principles of sustainable manufacturing.

4:40pm **MA4-1-MoA-10 Investigation of Technologically Driven Compositional and Structural Changes, Mechanical Properties, and Alloying of Transition Metal Diboride Thin Films**, ***Viktor Sroba [viktor.sroba@liu.se]***, Linkoping University, Sweden, Slovakia **INVITED**

This presentation focuses on the investigation of technologically driven compositional and structural changes, mechanical properties, and on alloying of transition metal diboride (TMB_2) thin films. The films were synthesized using state-of-the-art technological approaches, and modern analytical methods, including scanning transmission electron microscopy (STEM) and computational approaches based on density functional theory (DFT), were used for a complex characterization.

TMB_2 thin films grown by direct current magnetron sputtering (DCMS) are an exciting group of nanocomposite materials with excellent mechanical properties, making them a potential candidate for hard coatings in the cutting tools industry. But there are various structural drawbacks that limit their high-temperature application potential, brittle character, and low oxidation resistance. This work provides several pathways to mitigate these drawbacks.

The main topic is understanding the influence of deposition parameters of high power impulse magnetron sputtering (HiPIMS) on the growth of TMB_2 films. One of the benefits of HiPIMS is a high degree of ionization of the sputtered species, however accompanied by a lower deposition rate. These ions can then be attracted towards the growing film using synchronized bias. This high-energy bombardment results in compositional and structural changes in the film. Advanced hybrid HiPIMS/DCMS co-deposition combines a high degree of ionization provided by HiPIMS and a high deposition rate of DCMS.

In the first part, a study of cross-ionization in hybrid HiPIMS/DCMS co-deposition configuration was performed. It was demonstrated that the cross-ionization of the DCMS flux by HiPIMS is influenced by a relative ratio of two crucial parameters - ionization potentials and masses of the sputtered elements. In the second experimental part of the work, high ionization of the sputtered species during HiPIMS was used to suppress the formation of the amorphous boron-rich tissue phase of understoichiometric ZrB_x films while improving mechanical properties and oxidation resistance in the process. The next part of the research focused on the bombardment by the HiPIMS-generated W-ions showed significant densification of the TiB_2 films' structure and improved their mechanical properties, eliminating the need for external heating of the substrates during deposition. Lastly, on the basis of DFT predictions,alloying of TaB_2 and ZrB_2 films by aluminum and niobium, respectively, using conventional DCMS or hybrid DCMS/HiPIMS co-deposition, showed possible pathways to maintain or even improve mechanical properties at elevated temperatures and improvement in ductility.

Protective and High-temperature Coatings
Room Palm 3-4 - Session MA3-1-TuM

High Entropy and Other Multi-principal-element Materials I

Moderators: Frederic Sanchette, Université de Technologie de Troyes, France, **Frédéric Schuster**, CEA, France

8:00am **MA3-1-TuM-1 A Combinatorial Approach to Develop Sputter-Deposited Lanthanide-Containing High Entropy Alloys for ICF Applications**, ***Daniel Goodelman [goodelman1@llnl.gov]***, Lawrence Livermore National Laboratory, USA; *Minsuk Seo,* Lawrence Livermore National Laboratory, Republic of Korea; *Gregory Taylor, Alison Engwall-Holmes, Swanee Shin, David Strozzi, Brandon Bocklund, John Chesser, Jimmy Aut, Sergei Kucheyev, Leonardus Bimo Bayu Aji,* Lawrence Livermore National Laboratory, USA

In indirect-drive inertial confinement fusion (ICF) experiments at the National Ignition Facility (NIF), the hohlraum plays a critical role in achieving increased implosion yield, as it drives the fuel capsule's compression. Our simulations with the radiation hydrodynamics code LASNEX suggest that the fusion yield can be improved by using hohlraums made of high entropy alloys (HEAs) containing rare-earth (RE) elements. Here, we present results from a systematic study using combinatorial radio-frequency magnetron co-sputtering to develop a family of Gd-Ta-W-Au-Bi coatings with properties favorable for ICF applications, including high electrical resistivity for consideration in magnetically-assisted ICF schemes. These results provide a framework for the future development of RE-HEA hohlraum materials.

This work was performed under the auspices of the U.S. DOE by LLNL under Contract AC52-07NA27344 and was supported by the LLNL-LDRD Program under Project No. 26-ERD-011.

8:20am **MA3-1-TuM-2 Lanthanide- and Actinide-Containing High-Entropy-Alloy Coatings for Inertial Confinement Fusion Hohlraums**, ***Leonardus Bimo Bayu Aji [bayuaji1@llnl.gov]***, Lawrence Livermore National Laboratory, USA

A hohlraum, centimeter-scale sphero-cylindrical heavy-metal cans with wall thicknesses of 10–100 µm, is a key component of indirect-drive inertial confinement fusion (ICF) targets, as they determine the x-ray drive that implodes the fuel capsule. Our simulations predict that hohlraums made from rare-earth-containing high-entropy alloys (RE-HEAs) or depleted-uranium-containing high-entropy alloys (DU-HEAs) can achieve significantly higher x-ray coupling efficiencies than the best-performing single-element hohlraums made from Au or DU. Here, we present our progress in developing sputter-deposited RE- and DU-HEAs with material properties compatible with ICF target fabrication processes.

This work was performed under the auspices of the U.S. DOE by LLNL under Contract DE-AC52-07NA27344 and LDRD project 26-ERD-011.

8:40am **MA3-1-TuM-3 Machine Learning Assisted Design of Complex and High Entropy Alloys by Hybrid Hipims/Pulsed Dc Pvd Process for Low Carbon Energy Applications in Extreme Environments**, ***Paul Foulquier [paul.foulquier@cea.fr]***, CEA-INSTN, France; *Frédéric Schuster, Ryma Haddad, Fanny Balbaud-Célérier,* CEA, France; *Jean-Philippe Poli,* CEA List, France; *Eric Monsifrot,* AZ Concept, France **INVITED**

Materials and Data sciences convergence is the new paradigm governing the discovery acceleration of new materials, always more complex and integrating, in a virtuous approach, new durability and sovereignty requirements. Every great transitions are concerned: towards a sustainable future and energies, towards frugal digital applications, towards a medicine of the future always more personalised. This dynamic is organised at the national level by the PEPR DIADEM, a program gathering universities and research institutes to shape and promote this trend.

To achieve these ambitious goals of discovery acceleration, the role of acceleration platforms and self-driving laboratories is more and more significant. If numerical simulation tools are always more coupled to artificial intelligence approaches and allow the realisation of custom-made materials, in reality things are often much more complex. Indeed, no material can be made without elaboration processes and any materials keeps the memory of its elaboration process.

In this race to the discovery acceleration of materials, artificial intelligence may play a significant role in always more complex synthesis and shaping processes proficiency. Any types of Materials are concerned by these new approaches: metals, polymers, ceramics, composites including the industrials sectors in which they are involved : mobility, energy, microelectronics, construction, health. Among these high impact generic processes for many industrial sectors, thin film deposition technologies play a key role, in particular driven towards excellence by constraints and specifications imposed by the requirements always more specific of microelectronics applications.

We will present developments for applications in extreme environments using a hybrid pulsed direct current/HiPIMS system with multiple cathodes. In particular, these developments are related to applications in nuclear reactors.

In particular, a new approach to discovering new HEA-type coatings for corrosion in molten salts for small modular reactors will be presented. We will explore the feasibility of Ni-Al-Cr-Mo alloys.

Combinatorial approaches made possible by instrumented multi-target PVD technologies, coupled to artificial intelligence allowing the extraction of inter-parametric relations between processes parameters, are at the heart of this study dedicated to the development of a hybrid pulsed-DC/HiPIMS PVD process digital twin for the deployment of complex coatings for extreme media.

9:20am **MA3-1-TuM-5 EELS Study of Fe–Co–Ni Phosphides electrocatalysts for Hydrogen Evolution Reaction**, ***Chun-Te Chiang [tony25477210@gmail.com]***, Southern Taiwan University of Science and Technology, Taiwan; *Yu-Min Shen,* National Dong Hwa University (NDHU), Taiwan; *Yu-Tsung Lin, Jow-Lay Huang,* National Cheng Kung University (NCKU), Taiwan; *Sheng-Chang Wang,* Southern Taiwan University of Science and Technology, Taiwan

Abstract

Hydrogen is widely recognized as a promising clean energy carrier, with the hydrogen evolution reaction (HER) serving as a crucial step in sustainable hydrogen production. Transition-metal phosphides have attracted considerable attention owing to their excellent electrical conductivity and catalytic activity. In this study, Fe–Co–Ni phosphides were synthesized via a solution-based precursor route followed by phosphorization, forming nanostructured multimetallic phases with uniform morphology. Transmission electron microscopy (TEM) revealed nanoscale features, while energy-dispersive X-ray spectroscopy (EDS) confirmed the homogeneous distribution of Fe, Co, and Ni.

Electron energy loss spectroscopy (EELS) played a central role in elucidating the electronic structure evolution of the catalysts. The spectra revealed distinct edges corresponding to O (538.5 eV), Fe (712.0 eV), Co (783.0 eV), and Ni (857.0 eV), clearly reflecting the hybridization between metal 3d and P 2p orbitals and providing direct evidence of charge redistribution among the transition-metal sites. These findings demonstrate the strong correlation between the local electronic configuration and catalytic performance.

Electrochemical analysis further confirmed that Fe–Co–Ni phosphides exhibit remarkable HER activity, requiring only -0.176 V overpotential to reach -10 mA cm^{-2}. The polarization (I–V) curves showed rapid current response and low activation energy, while a Tafel slope of 109 mV dec^{-1} indicated a favorable Volmer–Heyrovsky mechanism. Long-term chronoamperometric measurements verified excellent durability, and subsequent durability and accelerated cycling tests lasting up to 100 hours will be conducted to comprehensively evaluate structural and electrochemical stability under realistic conditions.

This work not only demonstrates the successful synthesis of multimetallic phosphides but also highlights EELS as a powerful tool for probing the structure–property relationship, offering valuable insights for the rational design of efficient and durable hydrogen electrocatalysts.

Keywords: NiCoFeP ; EELS; electrocatalyst

9:40am **MA3-1-TuM-6 Solid-State Synthesis and In-Situ XRD Analysis of Titanium-Based Composite Oxides for Lithium-Ion Battery Anodes and Application**, ***Guan-Hong Lin [m56144031@gs.ncku.edu.tw]***, *Hsing-I Hsiang,* National Cheng Kung University (NCKU), Taiwan; *Yu-Min Shen,* National Dong Hwa University (NDHU), Taiwan

The rational design of multi-component oxide systems provides an effective pathway to balance high capacity and structural robustness in lithium-ion battery anodes. In this study, TiO_2–SnO_2 composite solid solutions were synthesized via a controlled solid-state reaction to explore the interplay between structural evolution and electrochemical performance. Structural analyses (XRD and TEM) confirmed the partial incorporation of Sn^{4+} into the rutile TiO_2 lattice, accompanied by limited phase segregation into SnO_2-rich domains at higher Sn contents. The coexistence of solid-solution and segregated regions generated a nanoscale heterostructure that enhanced

Li^+ diffusion and mitigated volume fluctuation during cycling. Among the synthesized samples, ST1450 initially delivered the highest capacity of 635.5 mAh g^{-1} at 0.2 C, but gradually declined to 231.9 mAh g^{-1} after 100 cycles, corresponding to the lowest degree of phase segregation. Electrochemical impedance spectroscopy (EIS) and distribution of relaxation time (DRT) analyses revealed that coherent TiO_2/SnO_2 interfaces effectively facilitated charge-transfer kinetics while preserving mechanical integrity. The optimized ST1450 sample exhibited an extremely low charge-transfer resistance of 3.85 Ω, reflecting improved electronic transport pathways. Furthermore, in-situ XRD measurements directly captured phase transitions during lithiation and delithiation, providing crucial insight into the dynamic reaction mechanism. The observed spinodal decomposition within the TiO_2–SnO_2 system forms a self-organized nanoscale microstructure that reinforces both ionic transport and structural stability. These results elucidate the lithiation pathway of TiO_2–SnO_2 composite oxides and highlight spinodal decomposition as a promising strategy for developing structurally adaptive, high-performance oxide anodes for next-generation lithium-ion batteries.

Keywords: **Composit**e **oxides**, **Anode materials**, **Fast-charging materials**, **In-situ XRD**, **Solid-state reaction**

Protective and High-temperature Coatings
Room Town & Country D - Session MA4-2-TuM

Boron-containing Coatings II

Moderators: Martin Dahlqvist, Linköping University, Sweden, **Anna Hirle**, TU Wien, Austria

8:00am **MA4-2-TuM-1 Tuning Structure and Mechanical Properties of TaB_x Films using HiPIMS***, Kateryna Smyrnova* ***[kateryna.v.smyrnova@gmail.com]****, Tomáš Roch, Martin Truchlý,* CENAM FMPI, Comenius University in Bratislava, Slovakia*; Peter Švec,* Institute of Physics, SAS, Slovakia*; Rainer Hahn, Helmut Riedl,* TU Wien, Austria*; Leonid Satrapinskyy,* CENAM FMPI, Comenius University in Bratislava, Slovakia*; Viktor Šroba,* Linkoeping University, Sweden*; Marián Mikula,* CENAM FMPI, Comenius University in Bratislava, Slovakia

Both experimental and theoretical studies have reported TaB_x to be promising for high-temperature and wear-resistant applications due to its exceptional hardness and oxidation resistance. However, achieving dense, nanocrystalline TaB_x with controlled stoichiometry remains a challenging task. To date, TaB_x films have been deposited only by conventional magnetron sputtering and high target utilization sputtering. This study demonstrates the pressure-controlled structure transformation of TaB_x films deposited by HiPIMS.

TaB_x coatings were deposited from a TaB_2 target in an Ar atmosphere at 340 °C using HiPIMS. Three groups of films were grown under three pressures (0.3 – 0.9 Pa) and two substrate bias conditions. The target current density was maintained at 1 A/cm^2 by adjusting the pulse frequency. The microstructure was analyzed using XRD and TEM. TaB_x films exhibited an amorphous structure at low pressure, a nanocomposite nature at moderate pressure, and a dense nanocrystalline structure at high pressure. This work presents the first systematic investigation into how energy flux controlled by pressure influences phase evolution in TaB_x deposited by HiPIMS. Cross-sectional SEM confirmed uniform thicknesses and sufficient adhesion. The chemical composition, as determined by WDS, showed that the B/Ta ratios rose from 1.05 to 1.4. This might be attributed to the reduced resputtering of B by thermalized reflected Ar neutrals at higher pressure. The nanoindentation method yielded a nanohardness of 26.2 GPa and Young's modulus of 344.6 GPa for amorphous films, improving to 42.4 GPa and 469.8 GPa for nanocrystalline ones. Fracture toughness (K_{IC}) was also determined by cantilever fracture testing in SEM.

These results establish working pressure in HiPIMS as a powerful parameter for controlling the crystallinity, stoichiometry, and mechanical properties of refractory borides. This study clarifies how changes in microstructure affect the densification and hardening of boron-rich films. The demonstrated ability to obtain dense, hard, and tough TaB_x films by HIPIMS highlights its potential for preparing next-generation materials for extreme environments. This work was funded by the Slovak Research and Development (No. APVV-24-0038) and the European Union under grant agreement No. 101158464 (COLOSSE).

8:20am **MA4-2-TuM-2 Solid Self-Lubrication Mechanism of B_2O_3 in Boride Based Thin Film Materials Under Various Atmospheres***, Daniel Pölzlberger* ***[daniel.poelzlberger@tuwien.ac.at]****,* Institute of Materials Science and Technology, TU Wien, Austria*; Norma Salvadores Farran, Tomasz Wojcik, Philip Kutrowatz, Rainer Hahn,* Christian Doppler Laboratory for Surface Engineering of high-performance Components, TU Wien, Austria*; Eleni Ntemou, Daniel Primetzhofer,* Department of Physics and Astronomy, Uppsala University, Sweden*; Carsten Gachot,* Institute of Engineering Design and Product Development, Research Unit Tribology, TU Wien, Austria*; Helmut Riedl,* Institute of Materials Science and Technology, TU Wien, Austria

Transition metal borides (TMBs) represent a highly promising family of materials for functional coatings due to their abundance, cost-effectiveness, and exceptional mechanical properties. These include a high melting point, hardness of up to 60 GPa, excellent chemical and thermal conductivity, and outstanding resistance to wear. Their distinct self-lubricating behavior arises from the formation of boron oxide, which reduces friction, although the fundamental mechanisms behind this tribo-reaction remain not fully understood, particularly for TMB-based thin films. Temperature and atmosphere play a significant role in boron oxide formation, making it essential to understand the oxidation behavior of physical vapor-deposited boride based films to interpret their friction and wear performance at elevated temperatures. Tribological tests reveal that sputter-deposited super-stoichiometric $TiB_{2.9}$ exhibits a significantly lower friction coefficient (~0.3) than sub-stoichiometric $TiB_{1.5}$ (~0.4) at 500°C, attributed to the higher oxidation rate of $TiB_{2.9}$ and the presence of B-rich tissue phases at column boundaries. Similarly, α-$WB_{2\pm z}$ coatings display self-lubrication via boron oxide formation, although growth related sub-stoichiometry limits boron availability during oxidation. The surrounding atmosphere further influences friction behavior: in oxygen-depleted conditions, oxidation and lubrication are minimal, whereas under humid conditions, water vapor reacts with boron oxide to form boric acid with layered, lubricious properties. To investigate these processes, we conducted ex-situ and in-situ oxidation studies of the TMB based thin films using elastic backscattering spectrometry (EBS) over a temperature range from room temperature to 940°C. The oxidation data, correlated with tribological, X-ray photoelectron spectroscopy, X-ray diffraction, and transmission electron microscopy analyses, provide new insights into the temperature-dependent lubrication mechanisms of boron oxide in TMB thin films.

8:40am **MA4-2-TuM-3 Super-Ordered MAB Phases: Theoretical Design of Novel Boron-Containing Materials with Simultaneous in-Plane and Out-of-Plane Chemical Ordering***, Martin Dahlqvist* ***[martin.dahlqvist@liu.se]****, Johanna Rosen,* Materials Design Division, Linköping University, Sweden

Chemical ordering in layered materials, such as MAX and MAB phases, enables precise tailoring of functional properties through structural motifs like in-plane (*i*-MAB, *i*-MAX) and out-of-plane (*o*-MAB, *o*-MAX) configurations. This study introduces super-ordered MAB phases (*s*-MAB), a novel structural material combining simultaneous in-plane and out-of-plane chemical ordering, previously unreported in synthesized MAB phases. Using first-principles density functional theory, we systematically investigated the thermodynamic stability of hypothetical quinary *s*-MAB phases with 314 ($M1_4M2_2M3_3Al_3B_{12}$) and 416 ($M1_4M2_2M3_6Al_3B_{18}$) compositions (M1, M3 = transition metals; M2 = Sc, Y) against a comprehensive set of competing phases, including ternary and quaternary MABs, disordered configurations, and binary/ternary borides at 2000 K. Our calculations predict 27 thermodynamically stable *s*-MAB compositions, predominantly featuring M1 = Cr, Mo, W, Mn, Fe and M3 = V, Nb, Ta, with M2 = Sc or Y promoting the combined in-plane and out-of-plane order. Notably, 416 structures exhibit conditions favoring partial or complete disorder, highlighting the nuanced stability landscape. These *s*-MAB phases offer a theoretical roadmap for synthesizing novel boron-containing compounds with precisely controlled atomic arrangements, potentially yielding unique mechanical, thermal, and electronic properties. Additionally, stable *s*-MABs can serve as precursors for 2D boridene (MBene) synthesis, expanding compositional flexibility for property tuning compared to existing boridenes. This work establishes a framework for designing advanced boride-based materials, with implications for high-temperature coatings, structural components, and electronic devices. Future experimental efforts should prioritize optimizing synthesis conditions to validate predicted chemical ordering and explore application-driven property enhancements.

Protective and High-temperature Coatings
Room Town & Country A - Session MA2-1-TuA

Hard and Nanostructured Coatings I

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

1:40pm **MA2-1-TuA-1 The Fabrication, Microstructure, and Characterization of Functional Electroless Ni-P Composite Surface Coatings on Dried Luffa as Bio-Plate***, **Tzu-Hsiu Hung [andybenny2012@gmail.com]**, Kai-Tse Tsai, Fan-Bean Wu,* National United University, Taiwan

This study employed luffa sponge as a natural substrate for the electroless deposition of nickel-phosphorus, Ni-P, alloy to enhance its structural performance. The phosphorus contented the Ni-P coating was controlled by adding sulfuric acid to adjusting the pH value of the plating bath, allowing analysis of compositional and property variations. The luffa sponge was first sensitized and activated using $SnCl_2$ and $PdCl_2$, followed by deposition in a solution containing $NiSO_4$, NaH_2PO_2, and $Na_2C_4H_4O_4$, with sulfuric acid used to adjust pH. Sodium hypophosphite acted as the reducing agent, promoting the co-deposition of Ni and P. Since the electroless plating rate was approximately 0.1–0.3 μm/min, electroplating was subsequently applied to increase film thickness and investigate its microstructure and mechanical behavior. This technique demonstrates a promising route for the functional surface modification of natural porous materials, enabling the fabrication of lightweight, high-strength composites with potential applications in electronic, structural, and environmental fields.

2:20pm **MA2-1-TuA-3 Characteristics of TiBCN-based Thin Film with Different Mo Content by Direct Current Plasma Chemical Vapor Deposition***, **Takeyasu Saito [t21165j@omu.ac.jp]**, Rizu Kurogi, Noki Okamoto,* Osaka Metropolitan University, Japan

Recently, the concept of high-entropy alloys (HEAs) has been extended from metallic systems to ceramic compounds such as nitrides, carbides, and borides, offering a promising strategy to develop next-generation protective thin films with superior hardness, thermal stability, and oxidation resistance. Ti-based thin film such as TiN, TiC, and TiCN was widely used as conventional protective thin films. Further improvements in hardness and oxidation resistance were carried out by incorporating elements such as B, to from TiBCN thin films composed of Ti(C,N) nanocrystals in an amorphous TiBCN matrix. Incorporating refractory metals such as Mo into Ti-based systems is also expected to enhance solid-solution strengthening and oxidation resistance due to their high melting points and in oxide formation resistance. However, the effects of Mo addition in multi-component hard thin films remain unclear. Most previous studies employed physical vapor deposition (PVD) methods such as magnetron sputtering or arc evaporation, while plasma enhanced chemical vapor deposition (CVD) provides potential advantage on higher conformality, stronger interfacial adhesion strength and low temperature fabrication for complex-shaped tools and components fabrication. The objective of this study is to investigate the role of Mo addition in TiBCN thin film containing Ti(C,N) nanocrystals and amorphous TiBCN, as well as the role of Mo addition on solid-solution hardening and oxidation resistance.

TiBCN thin films with different Mo contents were deposited on Si and cemented carbide (WC–Co) substrates using direct current (DC) plasma CVD at around 600℃ where WC–Co substrates were pretreated with aqua regia to improve interfacial adhesion. The precursor gases were $TiCl_4$, CH_4, N_2, BBr_3, and $MoCl_5$. It was confirmed that, B content in TiBCN films increased with increasing BBr_3 flow rate. The effects of Mo incorporation on phase formation, microstructure, and mechanical properties were systematically evaluated using X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), and nanoindentation. The results from the TiBCN film with Mo will be discussed in order to demonstrate the HEA design concept thin film by DC plasma CVD, which provides a promising method for low temperature next generation hard thin film procedures to enhance mechanical and chemical durability.

2:40pm **MA2-1-TuA-4 CrAlN-based Protective Nanostructured Coatings: Process–Structure–Property Correlations and Performance in Energy-Related Applications***, **Juan Carlos Sanchez-Lopez [jcslopez@icmse.csic.es]**, Teresa Cristina Rojas,* Institute of Materials Science of Seville (ICMS), Spanish National Research Council (CSIC), Spain*; Ramón Escobar-Galindo,* Universidad de Sevilla (US), Spain*; Santiago Dominguez-Meister, Marta Brizuela,* TECNALIA, Basque Research and Technology Alliance (BRTA), Spain*; Sonia Mato, Francisco Javier Pérez,* Universidad Complutense de Madrid (UCM), Spain **INVITED**

Chromium aluminum nitride (CrAlN)-based coatings represent a model system for understanding and engineering protective nanostructured materials operating under extreme environments. Over the past decade, our research has focused on correlating deposition parameters, microstructure, and functional performance of magnetron-sputtered CrAlN coatings. Particular attention has been paid to the influence of multiple factors, such as film architecture (single- or multilayered), aluminum and dopant concentrations and distributions (Y, Zr, Si, and O), the nature of the substrate, and the type of surrounding atmosphere (air or steam), on oxidation mechanisms, thermal stability, and functional behavior at high temperatures. These studies reveal how nanoscale architecture and compositional design govern mechanical integrity and resistance to degradation at high temperature. Complementary investigations have addressed tribological behavior and oxidation resistance in steam atmospheres, representative of demanding industrial and energy-generation conditions.

Building on this knowledge, recent developments have extended CrAlN-based coatings toward advanced energy technologies, including concentrated solar power systems, where coatings must combine optical functionality with long-term durability above 550 °C. This invited talk will review the evolution of CrAlN-based protective coatings from model hard systems to multifunctional materials for energy-related applications, emphasizing the process–structure–property relationships and degradation mechanisms that underpin their outstanding performance in harsh and sustainable operating environments.

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4:00pm **MA2-1-TuA-8 Enhancement of Thermal Stability of Sputtered Nanotwinned Ag Thin Films by Cu Doping for Reliable Electronic Applications***, **Jun-Hui Qiu [junhui-qiu@gapp.nthu.edu.tw]**,* Department of Engineering and System Science, National Tsing Hua University, Taiwan*; Yu-Lin Liao,* College of Semiconductor Research, National Tsing Hua University, Taiwan*; Fan-Yi Ouyang,* Department of Engineering and System Science, National Tsing Hua University, Taiwan

The rapid expansion of artificial intelligence, data centers, and electric vehicles has intensified the demand for reliable interconnect materials capable of withstanding high-temperature and high-current operating conditions. Silver (Ag) is a promising candidate for electronic applications due to its exceptional electrical conductivity; however, its poor thermal stability often leads to reliability concerns. To address this issue, this study investigates the effect of copper (Cu) doping on the thermal stability of sputtered nanotwinned Ag thin films.

Nanotwinned Ag and co-sputtered 6 at% Cu-doped Ag thin films were fabricated via magnetron sputtering and subsequently annealed at 200 °C, 300 °C, 400 °C, and 500 °C under a vacuum of 5.5 mTorr. In pure Ag films, the nanotwinned structure remained stable up to 400 °C for 1 hour but progressively transformed into large grains with increasing annealing time. After 48 hours at 400 °C, nanotwins disappeared completely due to abnormal grain growth, resulting in a (200)-oriented microstructure. In contrast, the 6 at% Cu-doped Ag films exhibited significantly slower grain coarsening and superior structural stability even after prolonged annealing.

At 400 °C, nanotwins were still observed after both 1 hour and 24 hours of annealing. Cu doping also suppressed the orientation transition from (111) to (200), indicating enhanced thermal stability. Furthermore, Cu addition increased hardness from 1.29 GPa to 3.33 GPa through solid-solution strengthening, while causing only a slight rise in electrical resistivity from 2.03 μΩ·cm to 2.90 μΩ·cm. These findings demonstrate that Cu doping effectively improves the thermal and mechanical stability of nanotwinned Ag thin films, providing a promising pathway toward reliable metallic interconnects for next-generation electronic devices.

4:20pm **MA2-1-TuA-9 Backscattered Argon Neutrals: Hidden Architects of Hf–Al–N Nanostructure Evolution***, **Naureen Ghafoor [naureen.ghafoor@liu.se]**, Marcus Lorentzon,* Linköping Univ., IFM, Thin Film Physics Div., Sweden*; Rainer Hahn,* TU Wien, Austria*; Diederik Depla,* Ghent University, Belgium*; Justinas Palisaitis, Jens Birch,* Linköping Univ., IFM, Thin Film Physics Div., Sweden

Transition metal aluminum nitrides (TM–Al–N) are multifunctional ceramics whose nanostructure can be tailored for extreme mechanical performance. However, the Hf–Al–N system remains largely unexplored. Here, we demonstrate that energetic backscattered argon neutrals, inherently produced during magnetron sputtering of heavy targets, dominate the structure formation in $Hf_{1-x}Al_xN_Y$ thin films—surpassing the influence of both ion assistance and substrate temperature.

Single-crystal cubic (c) $Hf_{1-x}Al_xN_Y$ films with $x < 0.30$ deposited on MgO(001) exhibit a unique three-dimensional checkerboard superstructure composed of HfN- and AlN-rich nanodomains aligned along the <001> directions. This periodic modulation, detected via XRD satellites and high-resolution STEM, originates from sub-surface spinodal decomposition triggered by backscattered Ar neutrals with energies exceeding the displacement threshold (~40–50 eV). SDTrimSP and SIMTRA simulations reveal that ~40 % of the neutrals impinging on the film surface possess sufficient energy to induce bulk atomic displacements, leading to compositional modulations even at low growth temperatures (300 °C). In contrast, varying ion flux (up to 15 ions per atom at 20 eV) or substrate temperature (300–900 °C) produced negligible structural changes, underscoring the primary role of energetic neutrals.

The superstructure period increases linearly with Al content (9–13 Å), correlating with hardness enhancements from 26 GPa for HfN_Y to ~38 GPa for c-$Hf_{1-x}Al_xN_Y$ due to dislocation pinning by strain fields. For Al-rich compositions ($x > 0.41$), a nanocrystalline wurtzite phase with 0001 texture forms, yielding ~22 GPa hardness. Micropillar compression of c-$Hf_{0.93}Al_{0.07}N_{1.15}$ confirmed brittle fracture along {110}<011> slip systems, reflecting superstructure-induced dislocation confinement.

These findings reveal a previously unrecognized mechanism of film nanostructuring in heavy-element sputtering: backscattered neutral atoms act as energetic sculptors, promoting coherent superstructure formation and enhanced mechanical performance. This insight extends beyond Hf–Al–N, providing a general framework for controlling nanostructure evolution in metastable nitrides through energetic neutral engineering.

4:40pm **MA2-1-TuA-10 From Grain Refinement to Precipitation Hardening: Si-Driven Microstructural Control in (Al,Mo,Ta,V,W)C Coatings***, M.A. Altaf, Balint Hajas,* TU Wien, Austria*; Szilard Kolozsvári,* Plansee Composite Materials GmbH, Germany*; Tomasz Wojcik, Alexander Kirnbauer, **Paul Mayrhofer [paul.mayrhofer@tuwien.ac.at]**,* TU Wien, Austria

High-entropy carbides (HECs) are emerging as promising candidates for extreme-environment applications due to their exceptional hardness and thermal resistance. This work investigates the influence of Si incorporation (0, 1, and 7 at%) on the microstructure, mechanical properties, and thermal stability of reactively sputtered $(Al,Mo,Ta,V,W)C_Y$ coatings. X-ray diffraction and transmission electron microscopy (TEM) confirm single-phase face-centered cubic structures for all compositions. Increasing Si content strongly refines the columnar grain morphology—from ~500 nm in the Si-free coating to ~20 nm in the 7 at% Si variant—and induces a pronounced (200) texture.

Upon annealing at elevated temperature, Si segregates to column boundaries and forms coherent nanoscale SiC precipitates, as evidenced by HAADF-STEM and EDS mapping. These precipitates act as effective barriers to boundary motion and dislocation activity, stabilizing the microstructure against coarsening. Mechanical testing shows very high as-deposited hardness values of 41 GPa (Si-free), 39 GPa (1 at% Si), and 41 GPa (7 at% Si). After annealing, all coatings retain excellent mechanical performance, with the Si-rich variants exhibiting minimal hardness reduction.

These results demonstrate that controlled Si addition enables precipitation-driven microstructural stabilization in HECs through strong carbide-forming enthalpies and multi-element chemical/size mismatch—rather than configurational-entropy effects. This mechanism provides a robust pathway for designing thermally stable, ultrahard ceramic coatings for demanding applications such as aerospace, energy, and high-temperature manufacturing environments.

5:00pm **MA2-1-TuA-11 Influence of Interlayers on Thermal Stability and Abnormal Grain Growth in Co-Sputtered Nanotwinned Cu–Ag Alloy Thin Films***, **Ding-Peng Lin [teddy910106@gmail.com]**, Yu-Lin Liao, Fan-yi Ouyang,* National Tsing Hua University, Taiwan

As electronic and power devices operate under increasingly high temperatures and voltages, bonding and interconnect materials must exhibit superior thermal stability and reliability. Silver-based nano-twinned films provide excellent electrical and thermal conductivity but are prone to abnormal grain growth during annealing, which degrades performance. This study investigates the effect of titanium (Ti) and tantalum (Ta) interlayers on the thermal stability and grain growth behavior of co-sputtered nano-twinned Cu–Ag alloy thin films during annealing. All films were deposited by magnetron co-sputtering and annealed at 200 °C, 300 °C, 400 °C, 500 °C, and 600 °C for 1, 24, and 48 hours under a vacuum of 5.5 mTorr. In the Ti-interlayer samples, the nano-twinned structure remained stable up to 400 °C for 1 hour but disappeared after prolonged annealing due to abnormal grain growth and texture transition. In contrast, the Ta-interlayer samples preserved a stable nano-twinned (111) structure even after annealing at 400 °C for 48 hours and 500 °C for 1 hour. The Cu–Ag alloy films with Ta interlayers also retained higher hardness and lower resistivity after annealing, indicating reduced interdiffusion and structural degradation. These results demonstrate that Ta effectively enhances the thermal and microstructural stability of nano-twinned Cu–Ag alloy thin films, establishing it as a superior diffusion barrier for high-temperature interconnect applications in next-generation power and electronic devices.

Protective and High-temperature Coatings
Room Palm 3-4 - Session MA3-2-TuA

High Entropy and Other Multi-principal-element Materials II

Moderators: Alexander Kirnbauer, TU Wien, Austria, **Pavel Soucek**, Masaryk University, Czechia

1:40pm **MA3-2-TuA-1 On the Structure and Properties of Refractory-Metal-Based High-Entropy Metal-Sublattice Ceramics***, **Alexander Kirnbauer [alexander.kirnbauer@tuwien.ac.at]**,* TU Wien, Thin Film Materials Science Division, Austria **INVITED**

The development of materials that can withstand high thermal and mechanical loads is in focus of many materials' science activities. In recent years high-entropy materials gained attraction of many researchers due to their vast compositional possibilities and therefore tuneable properties for many applications.

Here we show the beneficial effect of the high-entropy concept applied to several thin film material systems including borides, carbides, nitrides, and oxides. All the investigated coatings are based on refractory metals, including Cr, Hf, Nb, Ta, Ti, V, W, and Zr. The study focuses on the preparation by physical vapor deposition, their thermal stability and mechanical properties. All coatings investigated are comparably insensitive to the change of deposition parameters, such as reactive gas flow and bias potential. Additionally, they exhibit outstanding thermal stability and significantly retarded decomposition and softening processes, outperforming their commonly used binary or ternary constituents. Furthermore, we recently investigated the influence of He-ion irradiation on the mechanical properties of borides, carbides, and nitrides having the same metal sublattice. The results show that these coatings, if optimized regarding their structure, are possible materials to protect bulk materials from degradation due to the implantation of He.

2:20pm **MA3-2-TuA-3 Influence of Nitrogen Contents on the Microstructure, Mechanical, and Electrochemical Behaviors of AlCrNbSiTiMoNx high entropy alloy films deposited by HiPIMS***, **CHANG-YI JIANG [a910225y@gmail.com]**,* Department of Materials Engineering Ming Chi University of Technology, Taiwan*; Chia-Lin Li,* Center for Plasma and Thin Film Technologies, Taiwan*; Bih-Show Lou,* Chemistry Division, Center for General Education, Chang Gung University, Taiwan*; Jyh-Wei Lee,* Department of Materials Engineering Ming Chi University of Technology, Taiwan

High entropy alloy (HEA) films have attracted significant attention for applications in harsh environments due to their outstanding mechanical strength and excellent corrosion protection. In our earlier research, the increasing amount of Mo in the AlCrNbSiTiMo thin films can increase the hardness and significantly improve the corrosion resistance of thin films in harsh corrosive environments. Previous studies have demonstrated that nitrogen addition can significantly enhance the mechanical performance of HEA films effectively. In this study, AlCrNbSiTiMoNx HEA films with different nitrogen concentrations were deposited on silicon wafers, AISI 420, and 304 stainless steel substrates using a high power impulse magnetron sputtering (HiPIMS) system. The AlCrNbSiTiMoNx HEA films without nitrogen content exhibited a body-centered cubic (BCC) phase structure, while those with nitrogen contents between 15 and 26.3 at.% showed an amorphous structure. As the nitrogen content increased to 33.0 and 36.3 at.%, the films transformed into a face-centered cubic (FCC) nitride phase. Mechanical characterization revealed that the 36.3 at.% contained thin film achieved the highest hardness of 27.5 GPa. Potentiodynamic polarization tests demonstrated that the thin film with 33.0 at.% nitrogen exhibited a superior corrosion resistance, which is 319.3 times greater than that of 304 stainless steel. These findings indicate that AlCrNbSiTiMoNx HEA films possess excellent mechanical strength and corrosion resistance, underscoring their potential applications in harsh corrosive environments. The research provides valuable insights into the relationship between nitrogen content and the structural, mechanical, and electrochemical properties of AlCrNbSiTiMoNx HEA films.

KEYWORDS: (AlCrNbSiTiMo)Nx high entropy alloy thin films, high power impulse magnetron sputtering, mechanical properties, corrosion resistance

2:40pm **MA3-2-TuA-4 Development of CrMoNbWTi and CrMoNbTiWC high entropy alloy films by HiPIMS: effect of Ti and C contents***, Han-Chieh Chen, Chia-Lin Li, Bih-Show Lou, **Jyh-Wei Lee [jefflee@mail.mcut.edu.tw]**,* Ming Chi University of Technology, Taiwan

High entropy alloys (HEAs) and multicomponent alloys (MCAs) were first proposed independently by Professor Jien-Wei Yeh and Professor Brian Cantor in 2004. Since then, the research on HEAs and MCAs has been widely explored because of their unique properties, such as good mechanical strength and excellent corrosion resistance. Among the fabrication of HEAs, the HEA thin films have been studied extensively. In this work, we fabricated two series of HEA thin films. In the first series, CrMoNbW and Ti targets were co-sputtered using high power impulse magnetron sputtering (HiPIMS) technique to fabricate CrMoNbWTi films with different Ti contents ranging from 0 to 15.69 at.%. All CrMoNbWTi films exhibited a BCC structure. The 15.69 at.% Ti contained CrMoNbWTi film deposited at a Ti target power of 400 W showed excellent wear resistance, achieving a low wear rate of 1.3×10^{-6} $mm^3/N{\cdot}m$.

In the second series, CrMoNbTiWC thin films were fabricated using the same Ti target power of 400 W and with varying carbon contents via a plasma emission monitoring (PEM) feedback control by a HiPIMS deposition system. With increasing carbon content, the CrMoNbTiWC thin film structure transitioned from BCC to FCC and then to amorphous. Wear tests revealed that the T80 film, containing 83.1 at.% carbon, exhibited the lowest friction coefficient (COF = 0.16) and a lower wear rate of 2.1×10^{-7} $mm^3/N{\cdot}m$, demonstrating excellent wear resistance. Corrosion tests showed that the T20 film with 36.5 at.% carbon had superior corrosion resistance in 0.5 M H_2SO_4 (~1,500 times higher than 304SS), and in 3.5 wt.% NaCl (~33 times higher than 304SS). The detailed microstructure, mechanical, and corrosion properties of CrMoNbWTi and CrMoNbTiWC films with different Ti contents and varying carbon contents are comprehensively investigated in this study.

3:00pm **MA3-2-TuA-5 Synthesis and Characterization of Amorphous CrCuTaTiV High-Entropy Thin Films: The Role of Sputter Yield in Custom Target Design***, Uriel Cárdenas-Rojas, **Sandra E. Rodil [srodil@unam.mx]**, Carlos Ramos-Vilchis,* Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México

High-Entropy Alloys (HEAs) are a class of materials with exceptional properties driven by their high-entropy stabilized atomic structures. Since their introduction in 2004, HEAs have gained widespread attention because of their compositional versatility, which allows for designing materials with tailored properties for various applications. Thin film synthesis, especially through magnetron sputtering, is a powerful method for exploring new HEA compositions and coatings.

This work presents the synthesis and characterization of a new quinary HEA system, CrCuTaTiV, deposited as thin films using a single, custom-designed sputtering target. The target was created by combining five pure-element sectors, with each sector's area calculated based on its reported sputter yield to account for differences and achieve an equiatomic composition in the resulting film. Films were deposited at room temperature via DC magnetron sputtering with varying deposition times and powers (100, 200, and 300 W) to systematically examine how these parameters and sputter yields influence the final film composition and structure.

Comprehensive analysis confirms that all CrCuTaTiV coatings produced were amorphous. Compositional data showed that, despite the custom target design, the films had non-uniform compositions. Early in deposition, the composition was mainly affected by the element with the largest target area. Over longer deposition times, the composition became more uniform, settling at around 17-18 at% for Cr, Ti, and V, while Cu and Ta ranged from 20-30 at% (with some uncertainty due to Cu-Ta signal overlap). X-ray Photoelectron Spectroscopy (XPS) revealed the films had low oxygen content and retained a metallic nature, though surface analysis indicated a lower Cu presence and Ta enrichment at the very surface. These results provide important insights into the complex relationship between target design, deposition process, and compositional control in multi-element thin films.

4:00pm **MA3-2-TuA-8 Combinatorial Approach for the Synthesis of High-Entropy-Like Protective Nitride Coatings for Highly Aggressive Tribo-Corrosion Applications***, **Etienne Bousser [etienne.bousser@polymtl.ca]**, Olayinka Abegunde, Fellipy S. Rocha, Pedro Avila, Ludvik Martinu, Jolanta Ewa Klemberg-Sapieha,* Polytechnique Montréal, Canada

High-entropy alloys (HEAs) and high-entropy nitrides (HENs) have attracted increasing attention as protective coatings for demanding tribological and corrosive environments due to their high entropy, lattice distortion, and sluggish distortion effect. Compared to conventional transition-metal nitrides, HEN coatings can exhibit superior hardness–toughness, reduced wear, low friction coefficients, and enhanced corrosion resistance.

In this study, CrZrSiTiN coatings were deposited using a combinatorial magnetron sputtering process, generating a controlled compositional gradient across nine Ti6Al4V substrate samples deposited simultaneously. The depositions were carried out under a constant substrate bias, and a chromium sublayer was pre-sputtered to improve coating adhesion. The nitrogen flow rate was optimized to achieve a near-stoichiometric metal–nitrogen ratio (~1:1) characteristic of NaCl-type crystal structure nitride films.

Nanoindentation showed hardness values ranging from ~17 to 31 GPa and reduced moduli between ~138 and 282 GPa, depending on film composition. Tribo-corrosion testing in 1 M H_2SO_4 at open circuit potential (OCP) demonstrated that the most wear-resistant samples also exhibited the lowest coefficients of friction (~0.2), while sustaining the highest positive open circuit potentials (> 0.2 V) during sliding. Chromium-rich coatings with intermediate to low Si content, and reduced Ti and Zr concentrations outperformed the other compositions in terms of both wear and corrosion resistance. Wear volume analysis using optical profilometry revealed that all coatings significantly reduced wear compared to bare Ti6Al4V (by up to 260 times better), with the lowest wear rate of 1.3×10^{-6} $mm^3/N{\cdot}m$.

XRD revealed peak broadening, peak shifts, and texture variations, indicating lattice distortion, small grain size, and partial amorphization. Amorphized TiCrSiZrN coatings exhibited the best wear resistance and the highest recorded OCP values during and after wear. Samples with a dominant FCC (220) texture showed the second-best tribo-corrosion performance, whereas those with a dominant FCC (200) texture exhibited comparatively lower performance, but still significantly better than the bare substrate.

In conclusion, elemental composition plays a significant role in determining the preferred crystallographic texture of multi-element nitride coatings, which in turn governs tribo-corrosion performance. Lower concentrations of strong nitride-forming elements (e.g., Ti and Zr), combined with higher Cr content and adequate Si content, promote an amorphized microstructure with excellent wear and tribo-corrosion resistance.

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 $MPa \cdot m^{1/2}$ (average of both monolithic films) to 4.6 ± 0.3 $MPa \cdot m^{1/2}$ 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.

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

High Entropy and Other Multi-principal-element Materials III

Moderators: Frederic Sanchette, Université de Technologie de Troyes, France, **Pavel Soucek**, Masaryk University, Czechia

9:00am **MA3-3-WeM-4 CrMoNbTaV Refractory High-Entropy Alloy: From Bulk Material to Films via a Synergistic Theoretical–Experimental Approach***, Rafael Mendoza-Pérez, Ricardo González-Campuzano,* Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México*; David E. Martínez-Lara,* 2Escuela Nacional Preparatoria No.7 "Ezequiel A. Chávez", Universidad Nacional Autónoma de México*; Roxana M. Calderón-Olvera, Josué E. Romero-Ibarra, Ignacio A. Figueroa-Vargas,* ***Sandra E. Rodil-Posada [srodil@unam.mx]****,* Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México

High-entropy alloys (HEAs) have attracted significant attention since the initial findings by Cantor et al. and Yeh et al. in 2004. These alloys consist of five or more elements in equiatomic or nearly equiatomic proportions (5–35 at.%), resulting in high mixing entropy that reduces Gibbs free energy and promotes the formation of stable solid solutions. Their high configurational entropy originates from the multiple ways in which the different elements are arranged within the crystal lattice. This unique composition yields complex microstructures and outstanding mechanical properties, including corrosion resistance, high strength, toughness, and ductility. HEAs exhibit remarkable thermal stability and wear resistance, suggesting them as promising materials for multiple applications.

In this work, a synergistic theoretical-experimental approach was applied that simplified the planning, synthesis, characterization, and analysis of the atomic structure of custom-designed RHEA $Cr_8Mo_{25}Nb_{27}Ta_{16}V_{24}$composite coatings. The coatings were obtained by DC magnetron sputtering with a custom-designed RHEA target, meticulously produced through thermodynamic calculations, phase diagrams, and metallurgical processes. This ensured a single-phase, non-equiatomic composition that encompassed all the different predictors of the HEA. The samples were characterized using X-ray diffraction (XRD), Rietveld refinement, optical and mechanical profilometry, scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), high-resolution transmission electron microscopy (HRTEM), high-angle annular dark-field (HAADF), X-ray photoelectron spectroscopy (XPS), and atomic force microscopy (AFM). The findings confirm a predicted body-centered cubic (BCC) crystal structure in the coatings. Detailed atomic structural analysis by Rietveld refinement and HRTEM revealed a primary β-phase with a BCC crystal structure, coexisting with a minor β'-phase that surprisingly exhibited a body-centered tetragonal (BCT) crystal structure. These coatings demonstrated highly desirable properties: they remained flat, exhibited low oxidation, and reduced mechanical stress.

9:20am **MA3-3-WeM-5 Effects of HiPIMS Plasma Ionization and Deposition Parameters on the Microstructure and Mechanical Properties of TiZrNbTaMo High Entropy Alloy Films**, ***Chia-Lin Li* [chialinli@mail.mcut.edu.tw]**, Center for Plasma and Thin Film Technologies, Ming Chi University of Technology, Taiwan; *Bih-Show Lou,* Chemistry Division, Center for General Education, Chang Gung University, Taiwan; *Jyh-Wei Lee,* Department of Materials Engineering, Ming Chi University of Technology, Taiwan

TiZrNbTaMo high entropy alloys (HEAs) with a body-centered cubic (BCC) structure are well known for their excellent compressive yield strength and significant plasticity, which can be retained even in thin film form. These outstanding mechanical properties make them promising candidates for advanced applications. The deposition parameters play a critical role in determining the density, microstructure, and mechanical behavior of HEA thin films. In this study, TiZrNbTaMo high entropy alloy films (HEAFs) were deposited using high power impulse magnetron sputtering (HIPIMS), DC, and RF power sources to investigate the effects of deposition conditions on their structure and properties. HIPIMS, as an advanced physical vapor deposition (PVD) technique, enables a high degree of metal ionization and promotes dense film growth. To further understand plasma effects, the pulse frequency and duty cycle in HIPIMS were systematically varied while maintaining a constant average power. An ion meter was used to evaluate the degree of metal ionization under different peak discharge currents, and pulse-resolved optical emission spectroscopy (OES) was conducted to analyze the temporal evolution of excited species within each HIPIMS pulse, providing insights into discharge behavior and plasma–film interactions. The resulting films were characterized by X-ray diffraction (XRD) and transmission electron microscopy (TEM) to analyze their crystallographic structure and microstructure, while nanoindentation was used to measure hardness and elastic modulus. The TiZrNbTaMo HEAFs deposited by HIPIMS exhibited increased hardness due to the higher peak power density, which induced the coexistence of amorphous and nanocrystalline structures. This study demonstrates that combining HIPIMS deposition with pulse-resolved plasma diagnostics provides an effective approach to control plasma activation and tailor the microstructure and mechanical properties of TiZrNbTaMo high entropy alloy thin films, highlighting their potential for high-performance coating applications.

9:40am **MA3-3-WeM-6 Phase Formation, Microstructure and Selected Properties of Magnetron Sputtered Cr-Ta, Cr-Nb and Cr-v-Ta Thin Films**, *Jan-Ove Soehngen, Vincent Ott, Sven Ulrich,* ***Michael Stueber* [michael.stueber@kit.edu]**, KIT, Germany

Refractory alloy thin films, especially novel complex compositional and multiple principal element thin films, are of high interest in recent materials research. These materials can exhibit unique properties making them suitable candidates for a variety of high-load thermo-mechanical applications. Suprisingly, there is often a gap in the knowledge and data collection on thin film formation in more fundamental, simpler systems covering even binary or ternary refractory metals. In this study, we present results on phase formation, microstructure and selected properties on magnetron sputtered thin films in the systems Cr-Ta, Cr-Nb and Cr-V-Ta. The thin films were prepared by low-temperature, zero bias deposition from segmented targets to enable combinatorial studies of phase formation and microstructure evolution reflecting mainly the impact of the variation in chemical composition of the thin films. A major result is that by co-deposition from the segmented target single-phase solid solution b.c.c. structured thin films can be deposited in all systems under defined conditions. It is further of interest that the formation of a Laves phase, i.e. Cr_2Ta, Cr_2Nb or Art $(Cr,V)_2Ta$, can be suppressed by this approach. Finally, mechanical properties such as indentation hardness and modulus or electrical conductivity can be precisely controlled via tuning of the elemental composition of the thin films.

11:00am **MA3-3-WeM-10 Overcoming Strength-Plasticity Trade-Off in Complex Concentrated Alloy Thin Films by Engineering Their Atomic and Microstructure**, *Davide Vacirca, Arjun Curam,* Laboratoire des Sciences des Procédés et des Matériaux (LSPM) – CNRS, France; *Gregory Abadias,* Institut Pprime - CNRS - ENSMA - Université de Poitiers, France; *Andrea Li Bassi,* Nanolab, Departement of Energy, Politecnico di Milano, Italy; *Christian Ricolleau,* University of Paris, Laboratory of Matériaux et Phénomènes Quantiques, France; *Gerhard Dehm,* Max Planck institute for Sustainable Materials, Germany; ***Matteo Ghidelli* [matteo.ghidelli@lspm.cnrs.fr]**, Laboratoire des Sciences des Procédés et des Matériaux (LSPM) – CNRS, France

The design of high-performance structural materials is always pursuing the combination of mutually exclusive properties such as mechanical strength and plasticity. Complex concentrated alloys (CCAs) have recently attracted attention due to their superior mechanical properties, emerging from their multicomponent nature. However, such atomic complexity often prevents a nanoengineering approach with limited control over composition and microstructure, especially in bulk form.

Here, we exploit thin film (TF) synthesis to produce model FCC CCA-TFs with precise control over composition and microstructure (crystalline phase, density of structural defects and grain size), leading to large and tailored mechanical properties. Moreover, our approach encompassed both commonly employed synthesis method (i.e., sputtering) as well as pulsed laser deposition (PLD), leading to the development of novel nanostructures with unique nanoscale features [1].

Firstly, I will demonstrate a simple defect-engineering pathway in sputter-deposited CoCrNi CCA-TFs by introducing Fe to form Fex(CoCrNi)100–x [2]. Increasing the Fe content drives a structural transition from a dual FCC-HCP phase to a single FCC phase, accompanied by a decrease in defect density (stacking faults, nanotwins) and lattice distortion. This results in increased mass density and dislocation mobility, reflected by a decrease in hardness (from 9.6 down to 7.4 GPa), and increment in activation volume (up to ~13 b3).

Then, I will focus on CoCrCuFeNi CCA-TFs by PLD, with unprecedented microstructural control [3]. I will show how to synthetize ultrafine grain structures with controllable size (down to 12 nm) which can be further tailored by post-thermal annealing treatments, resulting in high hardness (11 GPa) and yield strength (2.0 GPa) due to Hall-Petch strengthening, outperforming similar CCA-TFs while maintaining high plasticity (no fracture at 30% strain). Moreover, these ultrafine CCA-TFs show remarkable thermal stability, with grain growth initiating only at 49% of the melting temperature, while maintaining high hardness (9.1 GPa) after annealing for 1h at 460°C.

Overall, we established a comprehensive nanoengineering strategy to tailor structure-property relationships in CCA-TFs, offering new opportunities to overcome the strength-plasticity trade-off.

[1] F. Bignoli et al., Acta Materialia, 300, 121456, (2025).[2] A. Curam et al., Submitted to Acta Mater. (2025). [3] D. Vacirca et al., Submitted to Materials Today, (2025).

11:20am **MA3-3-WeM-11 Exploring the Microstructure and Mechanical Properties of CoCrFeNiMn Thin Films**, ***Thomas Astecker* [thomas.astecker@tuwien.ac.at]**, TU Wien, Austria; *Peter Polcik,* Plansee SE, Austria; *Alexander Kirnbauer, Paul Heinz Mayrhofer,* TU Wien, Austria

Among high-entropy alloys, the equiatomic CoCrFeNiMn alloy, commonly known as the Cantor alloy, has emerged as a benchmark system due to its exceptional combination of strength, ductility, and thermal stability, stemming from its single-phase face-centered cubic structure and high-entropy effects. While the bulk properties of CoCrFeNiMn are well established, its behavior in thin-film form remains less explored, particularly under metastable synthesis conditions such as sputter deposition. In this work, we investigate the microstructure, thermal stability, crystal structure, and deformation mechanisms of CoCrFeNiMn thin films synthesized via magnetron sputtering. Films were deposited in an Ar atmosphere using a lab-scale PVD system at different substrate temperatures, with selected samples subjected to post-deposition thermal treatments. X-ray diffraction (XRD) was employed to assess crystal structure and phase formation, while mechanical behavior was probed using nanoindentation, in situ micropillar compression, and micro tensile testing, enabling direct comparison of plasticity and failure modes across multiple loading configurations. Chemical composition was analyzed by energy-dispersive X-ray spectroscopy (EDS), and transmission electron microscopy (TEM) provided insights into grain structure, defect evolution, dislocation activity, and potential deformation twinning. The results reveal the interplay between microstructure and mechanical response in sputtered CoCrFeNiMn thin films, demonstrating how microstructural features and size effects govern strength and ductility. These findings advance the understanding of deformation mechanisms in high-entropy alloys at small scales and inform their potential application as structural materials.

11:40am **MA3-3-WeM-12 Reactive Sputtering of CrMoNbWTiAgCx Carbide Films by High Power Impulse Magnetron Sputtering System: Effect of Ag and C Contents***, **BengYan Lu [w2859562@gmail.com]**, Yung-Chin Yang,* National Taipei University of Technology, Taiwan*; Chia-Lin Li,* Ming Chi University of Technology, Taiwan*; Bih-Show Lou,* Chang Gung University, Taiwan*; Jyh-Wei Lee,* Ming Chi University of Technology, Taiwan

High power impulse magnetron sputtering (HiPIMS) systems can produce thin films with dense microstructure compared with mid-frequency (MF) sputtering, due to the higher ion energy and plasma density. The combination of MF and HiPIMS has been reported to achieve higher deposition rates and reduced residual stress compared with HiPIMS alone. High entropy alloy (HEA) coatings, composed of multiple principal metallic elements forming carbides, borides, or nitrides, have attracted increasing attention for their exceptional mechanical and chemical stability.

In this study, CrMoNbTiWAg and CrMoNbTiWAgCx HEA carbide coatings were deposited using a superimposed HiPIMS–MF sputtering system. The Ag content was controlled by adjusting the power input to the Ag target, while the acetylene gas flow rate was tuned to control the degree of target poisoning during deposition. Microstructural evolution and phase formation were characterized using FE-SEM, XRD, TEM, and AFM, while mechanical properties such as hardness, adhesion, and wear resistance were evaluated by nanoindentation, scratch, and pin-on-disk tests. Electrochemical and oxidation behaviors were assessed via potentiodynamic polarization in 3.5 wt.% NaCl solution and thermogravimetric analysis (TGA) on X-750 superalloy substrates. Electrical properties were determined through four-point probe measurements, and antibacterial performance was evaluated via bacterial inhibition assays.

This study aims to elucidate the synergistic effects of Ag and C additions in improving the mechanical properties, corrosion protection, and multifunctional durability. The results are expected to provide valuable insights for developing durable and functional HEA carbide coatings through advanced HiPIMS technology.

Keyword: HiPIMS; high entropy alloy carbide; CrMoNbWTiAgCx coating; target poisoning; hardness; corrosion resistance.

Protective and High-temperature Coatings
Room Town & Country A - Session MA-ThP
Protective and High-temperature Coatings Poster Session

MA-ThP-1 Multienvironment Tribological Assessment of TiB_2:h-BN:a-C Coatings Deposited on 316L Stainless Steel*, **Ihsan Efeoglu [iefeoglu@atauni.edu.tr]**, Gokhan Gulten, Banu Yaylali, Mustafa Yesilyurt, Ali Emre, Yasar Totik,* Atatürk University, Turkey*; Justyna Kulczyk-Malecka, Peter Kelly,* Manchester Metropolitan University, UK

AISI 316L stainless steel is widely employed in mechanical and aerospace components; however, its tribological performance is often limited under dry and high-temperature sliding conditions. In this study, a solid-lubricating TiB_2:h-BN:a-C nanocomposite coating was deposited on 316L substrates using closed-field unbalanced magnetron sputtering (CFUBMS) with a hybrid HiPIMS + pulsed-DC power configuration. The coating architecture integrates hard TiB_2 domains for load support, h-BN for lamellar lubrication and thermal stability, and amorphous carbon for low shear and transfer-film formation. A Cr interlayer was introduced to improve interfacial bonding and accommodate residual stresses at the film–substrate interface. The tribological response of the coatings was evaluated under three different environments: dry sliding at room temperature, elevated temperature (300 °C), and boundary-lubricated contact with SAE 50 oil. Results demonstrated that the TiB_2:h-BN:a-C coating effectively reduced friction and wear compared with the uncoated steel, maintaining stable performance across varying temperature and lubrication regimes. Scratch testing further indicated strong adhesion and cohesive integrity. These findings confirm the effectiveness of TiB_2:h-BN:a-C nanocomposites as multifunctional protective coatings for extending the durability of stainless steel components operating under diverse tribo-mechanical conditions.

MA-ThP-2 Understanding Solid Particle Erosion in Multicomponent $Ti_{1-x}Al_xN$ Based Coatings Using Synchrotron Nanodiffraction*, **Anna Hirle [anna.hirle@tuwien.ac.at]**, Rainer Hahn, Philip Kutrowatz, Tomasz Wojcik,* Christian Doppler Laboratory for Surface Engineering of High-performance Components, TU Wien, Vienna, Austria*; Anton Davydok,* Helmholtz-Zentrum Hereon, Institute of Materials Physics, Hamburg, Germany*; Szilard Kolozsvári, Peter Polcik,* Plansee Composite Materials GmbH, Lechbruck am See, Germany*; Anders.O Eriksson, Carmen Jerg, Klaus Boebel,* Oerlikon Balzers, Oerlikon Surface Solutions AG, Balzers, Liechtenstein*; Helmut Riedl,* Christian Doppler Laboratory for Surface Engineering of High-performance Components, TU Wien, Vienna, Austria; Institute of Materials Science and Technology, TU Wien, Vienna, Austria

Protecting components operating in harsh environments, such as in energy production, aviation, and the tooling industry, is essential for ensuring sustainability and long service life. Solid particle erosion (SPE) occurs when high-velocity solid particles impact a material surface, leading to repeated mechanical damage and material loss. This phenomenon critically affects components such as turbine blades, compressor parts, and piping systems. A thorough understanding of SPE is therefore key to improving material durability and performance under erosive conditions. $Ti_{1-x}Al_xN$ protective coatings are widely applied due to their excellent oxidation, corrosion, and erosion resistance. However, increasing operational temperatures demand the development of new coating materials. Alloying $Ti_{1-x}Al_xN$ with Ta and Si has shown promising improvements in oxidation and corrosion resistance [1,2], yet their behavior under SPE conditions remains insufficiently understood.

The objective of the present study is to investigate the solid particle erosion resistance of $Ti_{1-x}Al_xN$ and $Ti_{1-x-y-z}Al_xTa_ySi_zN$ thin films. Accordingly, the coatings were deposited via cathodic arc evaporation using an industrial-scale Oerlikon Balzers INNOVA 1.0 system. SPE tests were performed employing a Jet Erosion Tester with corundum (Al_2O_3) particles at 70 m/s and impingement angles of 30° and 90°. Crater analysis by synchrotron nano-diffraction was employed to assess stress evolution induced by SPE, complemented by profilometry, scanning electron microscopy and transmission electron microscopy characterization. Both coatings demonstrate a significant reduction in mass erosion rates, with approximately 90% decrease compared to uncoated substrates. Synchrotron measurements reveal a clear influence of the as-deposited residual stress state on the stresses induced during erosion.

These results provide new insights into the interplay between residual stress and erosion-induced deformation in multicomponent $Ti_{1-x}Al_xN$ based coatings, contributing to the development of next-generation protective materials for high-temperature and erosive environments.

[1] X. Sun et al., Surf. Coat. Technol. 461 (2023) 129428.
[2] A.R. Shugurov et al., Vacuum. 216 (2023) 112422.

MA-ThP-3 Applicability of Mos2-Asic Heterostructure for Durable Supercapacitance and No2 Gas Sensing in Harsh Environment*, **Habeebur Rahman [habeeb.physics10@gmail.com]**, Davinder Kaur,* Indian Institute of Technology Roorkee, India

In the present work, the heterostructure of molybdenum disulfide (MoS_2)with amorphous silicon carbide (aSiC) on stainless steel (SS) and Si substrates was fabricated using a DC magnetron sputtering system. This unique heterostructure was examined for energy storage and NO_2 gas sensing applications suitable for harsh environmental conditions. The 2D MoS_2 nanostructured with dissolution resistive aSiC supercapacitor electrode delivers 1.5-fold enhancement in the gravimetric capacitance, a voltage window enlargement from 0.8V to 1.8 V, and an excellent stability of more than 4,000 charge-discharge cycles. Further, the high concentration NO_2 gas sensing performance of the MoS_2-aSiC on Si substrate revealed the stable and recoverable response at high operating temperatures. Therefore, loading aSiC with 2D MoS_2 enables durable electrode material for energy storage and NO_2 gas sensing applications in adverse conditions. The as-fabricated heterostructure was systematically studied by various material and electrochemical characterizations.

MA-ThP-4 Comparative Analysis of the Mechanical Properties of Layers Obtained in Three Different Steels by Atomic Diffusion of Boron.*, Enrique Hernández Sánchez,* **Luz Alejandra Linares Duarte [alejandra.linarespr@gmail.com]***, Diego Hernández Domínguez, Yesenia Sánchez Fuentes,* Instituto Politécnico Nacional, Mexico*; Raúl Tadeo Rosas,* Universidad Autónoma de Coahuila, Mexico*; José Guadalupe Miranda Hernández,* Centro Universitario UAEM Valle de México*; Rafael Carrera Espinoza, Melvyn Alvarez Vera,* Universidad de las Américas Puebla, Mexico*; Jonathan Jorge Ruíz Domínguez,* Instituto Méxicano de la Propiedad Industrial, Mexico

The boriding process was applied to generate hard layers on three different steels —AISI 1018, AISI 316L stainless steel, and AISI 4340 crankshaft steel —with varying treatment times and temperatures (2, 4 and 6h, and 900, 950, and 1000 °C). X-ray diffraction analyses were performed to characterize the phases formed in the coatings. Microscopy techniques were used to evaluate the morphology and thickness of the layers. In addition, micro- and nano-scale mechanical tests were performed to determine the change in the structural and functional properties of the steel. These steels, due to their nature, are subject to wear processes under operating conditions. Therefore, coating these steels with a hard layer that exhibits better properties could help them withstand the working conditions. The boride layers formed on the different steels exhibited varying morphologies. On the one hand, layers with a strong sawtoothed morphology formed on AISI 1018 steel. On the other hand, extremely flat layers were observed on AISI 316L stainless steel, and, finally, layers with a moderate front of growth were obtained on AISI 4340 crankshaft steel. The results indicated that not only were the layers' morphologies different, but also their chemical composition and mechanical properties changed. The layers obtained on AISI 1018 and the 4340 crankshaft steel were of monophasic Fe_2B nature with an 8.83% wt. of boron content, while the nature of those obtained on AISI 316L stainless steel was biphasic FeB/Fe_2B, with a 16.23% wt. of boron content. That difference in composition gives the layers different properties. The higher content of boron gives the biphasic layers the highest hardness, but also makes them more brittle than the monophasic layers. Therefore, it is essential to assess the characteristics of the resulting layers in relation to a specific application.

MA-ThP-5 Reactively Sputtered High-Entropy Metal-Sublattice Carbide Thin Films Based on Al-Cr-Nb-Ta-Ti*, **Thomas Astecker [thomas.astecker@tuwien.ac.at]**,* TU Wien, Austria*; Peter Polcik,* Plansee SE, Austria*; Alexander Kirnbauer, Paul Heinz Mayrhofer,* TU Wien, Austria

High-entropy metal-sublattice carbide thin films based on the equimolar Al-Cr-Nb-Ta-Ti system were synthesized by reactive magnetron sputtering in an acetylene-argon (C_2H_2-Ar) atmosphere. Films were deposited at varying reactive gas flow ratios and substrate temperatures between 450 and 650 °C, yielding single-phase rock-salt structured (fcc) solid solutions. Compositional analysis revealed the presence of an amorphous carbon phase and/or metallic sublattice vacancies. To explore the effect of silicon alloying, additional coatings containing 2.6, 6, and 8.8 at.% Si were prepared, which retained the single-phase crystal structure. The Si-free film exhibited an indentation hardness of 32.8 ± 0.4 GPa, decreasing slightly to

29.5 ± 1.1 GPa for the 8.8 at.% Si film, accompanied by a reduction in the average columnar grain width from 113 nm to 18 nm. No secondary phases formed during vacuum annealing up to 1000 °C for 15 min. While the unalloyed film softened significantly after the thermal treatment starting at 900 °C, the Si-containing coatings maintained their structural stability. Oxidation behavior in ambient air strongly depended on the Si content: at 800 °C, the Si-free film developed a thick (~7.8 µm) rutile-type oxide scale after 1 h, whereas the film with the highest Si-content formed only a dense, thin "passive" oxide layer, even after prolonged exposure at 1000 °C. These results highlight the pronounced beneficial effect of Si alloying on the thermal and oxidative stability of high-entropy carbide coatings, without compromising phase stability or mechanical properties.

MA-ThP-7 Synergistic Alloying Effects of Si and Y in Cr-Mn-Mo-N Thin Films: A Combined Experimental and DFT Study*, **Christian Gutschka [christian.gutschka@tuwien.ac.at]**,* TU Wien, Austria*; Lukáš Vrána, Matej Fekete,* Masaryk University, Czechia*; Zsolt Czigány,* Hungarian Academy of Sciences, Hungary*; Tatiana Pitoňáková,* Masaryk University, Czechia*; Katalin Balázsi,* Hungarian Academy of Sciences, Hungary*; Pavel Souček,* Masaryk University, Czechia*; Helmut Riedl-Tragenreif,* TU Wien, Austria

This study investigates the impact of Si and Y alloying on the microstructure and mechanical properties of Cr–Mn–Mo-based high- and medium-entropy nitride thin films. These films are fabricated by reactive DC magnetron sputtering, employing a combination of both ab initio calculations and experimental analysis. While the Cr–Mn–Mo–N system, in its pure form, displays a stable face-centred cubic (fcc) structure with a negative formation energy (E_f), the latter is known to decrease further with alloying with Si, Y, or both, thus enhancing thermodynamic stability. However, the process of alloying is accompanied by an increase in unit cell distortion, which in turn leads to the destabilization of the crystal structure – manifesting itself in amorphization of experimental thin films, with increasing alloying contents.

Chemical analysis of the grown films revealed that silicon and yttrium promoted nitrogen incorporation. However, complete stoichiometric metal-to-nitrogen ratios were not achieved, and systems with a considerable amount of nitrogen vacancies are formed. Similarly, simulations elucidated a prominent trend of nitrogen vacancies to relax alloy related cell distortions. Structural analyses confirmed the formation of a single-phase fcc solid solution presented here, with lattice expansion and crystallite size refinement being induced by increasing alloying concentrations. Furthermore, the simulations determined both nitrogen vacancy concentration and chemical composition to have a significant impact on ductility, with the highest levels of this property being observed at low nitrogen vacancy levels and when alloying with either Si or Y individually. These predictions showed a fair match with ductility estimates from experiment. More prominently, predictions of elastic properties from experiment and simulation agreed within error range, demonstrating that alloying has a beneficial effect on hardness without compromising the material's elasticity, with Si alloying alone achieving the highest hardness values of 20.5 GPa. An analysis of chemical bonding in the compounds found a synergistic sharing of nitrogen atoms between tetrahedral coordinated silicon species and yttrium atoms, mediated through N-vacancy introduction, as possible explanation for the good mechanical performance of the thin films.

MA-ThP-9 Thermal Stability and Mechanical Performance of Si-Modified High-Entropy (Al,Mo,Ta,V,W)C Coatings*, Muhammad Awais Altaf, **Balint Istvan Hajas [balint.hajas@tuwien.ac.at]**,* TU Wien, Institute of Materials Science and Technology, Austria*; Szilard Kolozsvari,* Plansee Composite Materials GmbH, Germany*; Tomasz Wojcik, Alexander Kirnbauer, Paul Heinz Mayrhofer,* TU Wien, Institute of Materials Science and Technology, Austria

High-entropy carbide (HEC) thin films (Al,Mo,Ta,V,W)C were reactively deposited on sapphire substrates with varying Si content to investigate its influence on microstructure, mechanical performance, thermal stability, and oxidation behavior. All coatings exhibit a single-phase face-centered cubic structure, with lattice parameters decreasing from ~4.34 Å for Si-free HEC to ~4.24 Å for the 7-at%-Si containing HEC-Si7, indicating preferential substitution of Si into the metallic sublattice, because fcc-SiC would have a lattice parameter of 4.35 Å. Si addition transformed the microstructure from coarse, columnar grains to smooth, dense, and near-amorphous surfaces, and shifted the preferred growth orientation from random to (200).

Nanoindentation revealed that as-deposited coatings exhibit hardness and elastic modulus of 30.7 GPa and 506 GPa for HEC, which decrease with increasing Si content to 28.0 GPa and 383 GPa for HEC-Si7. Upon vacuum annealing at different temperatures T_a, HEC degraded significantly, with hardness and modulus dropping to 14.4 GPa and 168 GPa for T_a = 1000 °C, whereas HEC-Si7 retained high mechanical stability, maintaining 26.3 GPa hardness and 381 GPa elastic modulus for T_a = 1000 °C and 24.2 GPa and 389 GPa for T_a = 1200 °C. Isothermal oxidation experiments at 600 and 800 °C for 1 h showed no protective oxide scale formation with or without Si, as already at 600 °C all coatings are oxidized through. However, the stability during vacuum annealing treatment demonstrate that controlled Si incorporation enhances microstructural integrity and mechanical robustness of these high-entropy carbide coatings, offering a promising route for high-temperature protective applications.

MA-ThP-10 Influence of the Ti/Al Ratio on the Performance of Ti-Al-N Coated Tools in the Machining of Stainless Steel 304*, **Felipe Batista dos Anjos [batista.anjos@pucpr.edu.br]**, Carlos Bernardo Gouvêa Pereira, Carlos Augusto Henning Laurindo, Fred Lacerda Amorim, Michelle Sostag Meruvia, Paulo Cesar Soares Junior, Ricardo Diego Torres,* Pontificia Universidade Católica do Paraná, Brazil

Machining austenitic stainless steel presents significant challenges due to various factors. One key issue is the material's low thermal conductivity, which can lead to thermal overload on the cutting tool. Additionally, these materials tend to adhere to the tool's cutting edge, resulting in adhesive wear and the formation of a built-up edge. To enhance tool performance, ceramic coatings can be applied, as they improve hot hardness and provide thermal and chemical insulation.

This study focused on evaluating the effect of the Ti/Al ratio in titanium, aluminum, and nitrogen-based coatings during the turning process of AISI 304 stainless steel. The coatings investigated were Ti0.56Al0.44N (TiAlN – Futura Nano®) and Ti0.37Al0.63N (AlTiN – Latuma®), both produced by Oerlikon Balzers Revestimentos Metálicos LTDA. Characterization of the coatings through Energy Dispersive X-ray Spectroscopy (EDS) revealed that the atomic ratio of Ti to Al in the Ti0.56Al0.44N coating is 1.27, while for the Ti0.37Al0.63N coating, the ratio is 0.59.

Machining tests were conducted on a CNC lathe equipped for cutting force acquisition, which was monitored over time until the end of the tool's life. To assess tool wear, the surface roughness of the workpiece was measured using a profilometer after each force test, and the tool geometry was analyzed using a scanning electron microscope (SEM). Preliminary results suggest that the tool coated with Ti0.37Al0.63N may exhibit lower cutting forces and a longer tool life compared to the Ti0.56Al0.44N coating. Additionally, nanoindentation tests indicated that the Ti0.37Al0.63N coating has a higher hardness than the Ti0.56Al0.44N coating, resulting in greater wear resistance.

Keywords: PVD Coatings, Tool Wear, Cutting Forces.

MA-ThP-13 Nitrogen-Dependent Structural and Mechanical Properties Evolution of $AlCrNbSiTiN_x$ High Entropy Alloy Nitride Coatings Deposited by HiPIMS*, Sheng-Jui Tseng,* National Taipei University of Technology, Taiwan*; **Jyh-Wei Lee [jefflee@mail.mcut.edu.tw]**,* Ming Chi University of Science and Technology, Taiwan*; Yung-Chin Yung,* National Taipei University of Technology, Taiwan*; Bih-Show Lou,* Chang Gung University, Taiwan*; Chia-Lin Li,* Ming Chi University of Science and Technology, Taiwan

High entropy alloy (HEA) nitride coatings have drawn significant attention owing to their excellent mechanical strength, corrosion resistance, and superior thermal stability. In this study, AlCrNbSiTiNx HEA nitride coatings were deposited on Si wafers, AISI 420, and 304 stainless steel substrates using high power impulse magnetron sputtering (HiPIMS). The effect of nitrogen content on the target poisoning behavior of the equimolar AlCrNbSiTi target was monitored and controlled through a plasma emission monitoring (PEM) feedback control system. Target poisoning ratios ranging from 10% to 90% were systematically examined to evaluate their influence on the microstructure and properties of the coatings. The nitrogen-free AlCrNbSiTi coating exhibited an amorphous structure, while the introduction of nitrogen promoted the formation of a face-centered cubic (FCC) nitride phase. Both the hardness and elastic modulus increased with nitrogen addition due to solid-solution strengthening and the formation of metal nitrides. Thermogravimetric analysis (TGA) conducted at 950 °C in air demonstrated that the AlCrNbSiTiNx coatings possessed excellent oxidation resistance. The relationship between nitrogen content, target poisoning ratio, mechanical properties, and oxidation behavior at 950 °C of the AlCrNbSiTiNx coatings was comprehensively studied in this work.

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