Protective and High-temperature Coatings
Room Town & Country A - Session MA1-1-TuM

Coatings to Resist High-temperature Oxidation, Corrosion, and Fouling I

Moderators: Justyna Kulczyk-Malecka, Manchester Metropolitan University, UK, **Francisco Javier Pérez Trujillo**, Universidad Complutense de Madrid, Spain

8:00am **MA1-1-TuM-1 High Temperature Corrosion Resistant Coatings: Recent Aluminide Developments for Renewable Energy Applications***, **Pauline Audigié [audigiep@inta.es]**, Cristina Lorente, Sergio Rodriguez, Loïc Oger, Alina Agüero,* Instituto Nacional de Técnica Aeroespacial (INTA), Spain **INVITED**

Protective coatings are known for many decades as first-rate mitigation methods to hinder high-temperature oxidation and corrosion in several industrial sectors. For many years, INTA has been developing diffusion coatings by spraying Al slurry onto different substrates such as different composition of Fe, Ni, Ti and Mo-based alloys. Recently, new developments have been focused on renewables energies including concentrated solar power (CSP) plants with thermal energy storage systems and hydrogen-fueled combustion systems. For those applications, some components require coatings that are resistant to corrosion/oxidation and mechanical stresses which might also give rise to considerable cost reduction by using lower cost alloys. In particular, new slurry aluminide coatings deposited onto TA6V and Ti6246 Ti-based alloys are being explored for compressor parts of aircraft engines and also onto 310H and 347H austenitic steels for molten nitrate and carbonate resistance in hot storage tanks and tubes in CSP plants. An overview of the global deposition process including surface preparation, deposition methods and thermal treatments will be shown for each generated coating. The coating formation mechanism and their prevailing protection mechanism in their respective corrosive environment will be presented. Furthermore, the loss of the protective oxide former element, Al in this case, by coating-substrate interdiffusion during exposure at high temperature can lead to premature degradation of the coating. Efforts have thus been pursued to reduce interdiffusion in the Ti6246 and 310H coated systems by incorporating Si particles in the slurry. This led to the formation of Si-Al rich diffusion barriers for which the latest progresses and corrosion results in both environments will be presented.

8:40am **MA1-1-TuM-3 Molten Salt Corrosion and Stress Corrosion Cracking Performance of Slurry Aluminide Coated Steels for Thermal Energy Storage***, **Loïc Oger [loge@inta.es]**, Pauline Audigié,* Instituto Nacional de Técnica Aeroespacial (INTA), Spain

Thermal energy storage systems associated to concentrated solar power (CSP) plants emerged as key technologies to allow consistent energy distribution while reducing electricity cost. However, their wide-range deployment is limited by the cost of the structural materials and by their short lifespan which imposes recurrent and expensive operation and maintenance. This is mainly due to the use of corrosive high-temperature molten salts as heat transfer fluid and to stresses generated by temperature variations. The European project COMETES was thus designed to develop coated materials as cost-effective alternatives suitable in current plants and in more aggressive operating conditions considered for next-gen CSP. The present study focused on molten salt corrosion and stress corrosion cracking (SCC) resistance of:

(1)A slurry aluminide coated P91 martensitic-ferritic steel considered as a promising alternative for current use in Solar Salt (60wt%$NaNO_3$ – 40wt%KNO_3) up to 580 ºC because of its 3 to 10 times lower cost than the currently used Ni-based materials.

(2)a newly developed coated 347H austenitic steel, capable to withstand higher operating temperatures – 700 ºC in the present study – with 32wt%Li_2CO3 – 33wt%Na_2CO_3 – 35wt%K_2CO_3 carbonate to achieve higher plant efficiency and increase the heat storage duration.

1000 h-hot corrosion study of the uncoated materials in their respective molten salt and temperature showed significant degradation of the substrates with the formation of various alkaline oxides. Tensile tests performed at a strain rate of 10^{-3} s^{-1} after such exposure showed a relatively low sensitivity of the P91 to SCC while a collapse of the 347H mechanical properties was evidenced. The slurry aluminide coated P91, composed by a homogeneous FeAl outer layer with Kirkendall pores formed in the interdiffusion zone, significantly increased the corrosion resistance when compared with the uncoated P91 in Solar Salt capable to withstand up to 10,000 h at 580 ºC. On the 347H, a 3-layers coating developed with several Fe-Al-Cr rich phases in the top layer. Despite the hardness heterogeneity (200 to 1000 $HV_{0.05}$) and the coating evolution along the exposure with the occurrence of phase transformations and Cr_3Si precipitation, aluminide coated 347H withstood at least 2,500 h in carbonatesat 700 ºC with the formation of a mixed γ/α-$LiAlO_2$ top layer. From the mechanical view, both steels were shown to have slightly lower maximal stresses after coating, which was attributed to crack initiation in the latter and then propagation into the substrate. Nonetheless both coatings efficiently increased the SCC resistance of the steels in molten salts.

9:00am **MA1-1-TuM-4 Prediction of the Ageing Behavior of Diffusion Aluminide Coatings Using Machine Learning***, **Vladislav Kolarik [vladislav.kolarik@ict.fraunhofer.de]**, Maria del Mar Juez Lorenzo,* Fraunhofer Institute for Chemical Technology ICT, Germany*; Pavel Praks, Ranata Praksová,* IT4Innovations National Supercomputing Center, VSB - Technical University of Ostrava, Czechia

Aluminide diffusion coatings provide an effective and cost-efficient solution for protecting steels from high-temperature corrosion in harsh environments. They can be applied as aluminum slurries through various deposition techniques, such as spraying or brushing, followed by heat treatment to create the diffusion layer. Machine learning algorithms show great potential for modeling and predicting the aging behavior of these coatings, while facilitating their optimization and customization. Machine learning relies solely on data and does not require physical models to describe dependencies. This is especially advantageous for systems influenced by multiple parameters, where the extent of each parameter's impact on the system is not well known. Symbolic regression and decision tree-based algorithms, such as XGBoost and Catboost were employed to explore the potential of machine learning in modelling the coating characteristic over time and forecasting the ageing behavior.

Data files for input were created collecting parameters, such as time, temperature, atmosphere, overall coating thickness, thicknesses of the partial layers, number of the partial layers, type of slurry etc. To evaluate the aging process, two key output parameters were modeled based on selected input variables: (1) the ratio of the outer Fe_3Al layer to the total coating thickness, and (2) the aluminum concentration within the outer Fe_3Al layer. The first parameter indicates the conditions under which the diffusion coating evolves into a single aluminum-poor layer—this occurs when the ratio equals 1. The aluminum concentration, on the other hand, reflects how much aluminum remains in the coating, which is essential for forming a protective alumina layer. Decision tree-based algorithms, such as XGBoost, are well-suited for assessing the degree of influence of individual parameters, with time emerging as the most significant factor, followed by the thickness of the aluminum-poor Fe_3Al layer.

The results demonstrate that machine learning is highly effective for analyzing complex material systems influenced by numerous parameters, where the relationships and significance of these parameters are challenging to capture through traditional physical modeling.

9:20am **MA1-1-TuM-5 AI-Enhanced Correlative Microscopy: A Multi-Modal Approach to Automotive Coating Evaluation***, **Hugues G. Francois-Saint-Cyr [hugues.fsc@thermofisher.com]**,* Thermo Fisher Scientific, USA*; Alice Scarpellini, Bartlomiej Winiarski,* Thermo Fisher Scientific, Netherlands*; Roger Maddalena, Rengarajan Pelapur,* Thermo Fisher Scientific, USA

The automotive industry extensively employs zinc-based coatings to enhance corrosion resistance and extend the lifespan of steel components exposed to harsh atmospheric conditions. These protective layers, applied through hot-dip galvanizing and electroplating, prevent oxidation of the underlying steel. The effectiveness of these coatings depends on thorough material quality assessment, adhesion evaluation, and stringent final product control.

Scanning electron microscopy (SEM), along with other advanced analytical characterization techniques, is crucial for detailed evaluation of zinc-based coatings. SEM supports correlative microscopy (CM) workflows, integrating imaging, analytical solutions, and AI-assisted image analysis to provide a multi-modal and multi-dimensional view of the materials under investigation. The site-specific use of Plasma Focused Ion Beam-SEM (PFIB-SEM) cross-sections enables highly targeted analysis, revealing detailed microstructural features and providing comprehensive compositional and crystallographic information.

The Apreo ChemiSEM exemplifies this approach by enabling comprehensive surface and cross-sectional analysis of both coatings and steel substrates. Its correlative capabilities combine imaging, energy dispersive X-ray spectroscopy (EDS) via ChemiSEM Technology, and electron backscatter diffraction (EBSD) with the TruePix detector. Cross-sectional analysis is performed on both metallographically prepared sections and PFIB-prepared cross-sections, providing detailed insights and accelerating the characterization of coating morphology and defect identification.

Integrating imaging with ChemiSEM Technology allows for detailed investigation of surface oxidation, inclusions, and inhibition layers within the coatings. The TruePix detector identifies areas of high dislocation density and other crystalline defects, offering a deeper understanding of material weaknesses that may correlate with reduced adhesion or other issues.

AI-assisted image analysis enhances the characterization process by significantly reducing the time required to interpret complex datasets. Deep learning models integrated into the workflow provide accurate, large-scale analysis of data collected at both micro- and nanoscale levels, enabling validation over millimeter-scale regions. This synergy between advanced microscopy and AI ensures a robust and comprehensive evaluation of zinc-based coatings, linking structure, processing, property, and performance in automotive applications.

9:40am **MA1-1-TuM-6 High-Temperature Corrosion in Contact with Molten Glass Improved by Thermal Spray Coating***, Michelle Hartbauer* ***[michelle.hartbauer@uni-bayreuth.de]****,* University of Bayreuth, Germany*; Thomas Dörflinger,* Neue Materialien Bayreuth GmbH, Germany*; Helge Schumann,* Wiegand-Glashüttenwerke GmbH, Germany*; Gilvan Barroso,* Rauschert Heinersdorf-Pressig GmbH, Germany*; Haneen Daoud,* Neue Materialien Bayreuth GmbH, Germany*; Florian Scherm, Uwe Glatzel,* University of Bayreuth, Germany

Glass manufacturing demands extreme conditions, exposing components to high-temperature corrosion through contact with molten glass at temperatures above 1100 °C. Thermal spray processes have emerged as a valuable solution for creating protective coatings that can significantly enhance material performance in such harsh environments. These coatings improve component longevity by increasing hardness, wear resistance, and corrosion resistance. Known for their performance, ease of application, and cost-effectiveness, thermal spray coatings are widely used across various applications.

To address these challenges, this study employs thermal spray processes to deposit an Al-Ni alloy onto a substrate of lamellar grey cast iron. Al and Ni wires are applied simultaneously via arc spray process. An electric arc generated between two consumable wires produces intense heat, melting the wire tips. Nitrogen gas then propels the molten material onto the substrate's surface, where it rapidly solidifies to form the protective coating. Heat treatment was then carried out.

To examine the coating's corrosion resistance, samples were immersed in molten glass for up to 48 h and subsequently prepared for analysis using metallographic procedures. Cross-sectional imaging allowed the analysis of the reaction zone between the thermal spray coating and molten glass.

Analyses of microstructure and chemical composition were conducted using SEM and energy-dispersive spectroscopy (EDS) across various stages: after thermal spraying, post-heat treatment, and after glass contact. Additionally, phase identification was carried out using X-ray diffraction (XRD).

Coating thicknesses with 100 - 300 µm were achieved. The differences in composition and heat treatment have a great influence on the microstructure of the thermal sprayed coatings. Differences in elemental distribution, phases formed, and their corresponding properties, particularly hardness, were observed.

10:00am **MA1-1-TuM-7 Microstructural Characterization and Isothermal Oxidation Behavior of a Nanolaminate Ti_2AlC MAX Phase Coating on TiAl 48-2-2***, Radosław Swadźba* ***[radoslaw.swadzba@git.lukasiewicz.gov.pl]****,* Łukasiewicz Research Network – Uppersilesian Institute of Technology, Poland*; Bogusław Mendala, Lucjan Swadźba,* Silesian University of Technology, Poland*; Nadine Laska,* German Aerospace Center (DLR), Germany*; Sarra Boubtane,* German Aerospace Center, Germany*; Dariusz Garbiec,* Łukasiewicz Research Network – Poznań Institute of Technology, Poland

This study investigates the application of the Closed Hollow Cathode Physical Vapor Deposition (CHC-PVD) method for depositing Ti_2AlC MAX phase nanolaminate coatings on a TiAl 48-2-2 alloy substrate. During deposition, samples were placed within an 80 mm-diameter, 160 mm-long hollow cathode using a target composition of Ti-25Al-25C (at.%). The resulting coatings, approximately 12 µm thick, exhibited a columnar microstructure. Advanced characterization techniques, including High-Resolution Scanning Transmission Electron Microscopy (STEM) and High-Resolution TEM (HRTEM), were employed to analyze the microstructure of both as-deposited coatings and coatings subjected to isothermal oxidation testing.

The oxidation resistance of the obtained coatings was evaluated using Thermogravimetric Analysis (TGA) at 850 °C for 20 hours in an air atmosphere. Mass change analysis revealed that the parabolic rate constant for the coated material was over five times lower than that of the uncoated substrate. Detailed STEM and HRTEM analyses showed Ti_2AlC nanolaminates within the columnar structures, with basal planes of the Ti_2AlC phase aligned parallel to the growth direction at the core and tilted in sub-columnar regions.

After the isothermal oxidation test it was found that a very thin and continuous alumina oxide scale is formed on the coated TiAl 48-2-2 with a thickness of around 320 nm. HRTEM and FFT (Fast Fourier Transform) imaging were applied to study the phase composition of the oxide scale and showed the presence of a mixture of transition θ-Al_2O_3 and stable α-Al_2O_3.

10:20am **MA1-1-TuM-8 Harnessing Ti_2AlN MAX Phase Based PVD Coatings on Titanium Aluminide Alloys for High Temperature Applications***, Sarra Boubtane* ***[sarra.boubtaneepzammouri@dlr.de]****,* German Aerospace Center, Germany

Nanolaminate coatings based on MAX phases (where M= Ti, A=Al, and X is nitrogen) exhibit a distinctive combination of ceramic and metallic material properties, offering considerable potential for utilization in high-temperature environments.

It is unfortunate that the application of MAX phases as coating material on various Ti- or Ni-based alloys results in degradation due to interdiffusion processes between the coating and the alloy, which is accompanied by an Al-depletion. A promising strategy involves combining a γ-TiAl substrate with a MAX phase coating, as the substrate can serve as an Al reservoir, replenishing the coating through outward diffusion of Al. This approach could also enhance the mechanical properties of such coated components compared to other protective but brittle intermetallic coatings on TiAl alloys.

In this study, a Ti_2AlN-MAX phase-based coating was deposited using reactive magnetron sputtering using pure elemental targets of Ti, Al and nitrogen as a reactive gas. The deposition process was studied using a variety of substrates, including inert Al_2O_3 and MgO substrates, as well as an already been used γ-TiAl alloy. This alloy TiAl48-2-2 (48Ti–48Al–2Nb–2Cr in at.%), supplied by GfE–Gesellschaft für Elektrometallurgie in Nuremberg, Germany, was utilized for all oxidation and interdiffusion experiments. The two-fold rotation ensures homogeneous deposition, with a thickness of 10 µm and near to the requisite stoichiometric composition of the Ti_2AlN MAX phase. Due to the low substrate temperature during deposition, the resulting layer was X-ray amorphous. A post-annealing treatment was performed at 800°C in a high vacuum furnace for one hour for crystallization. Additionally, high-temperature X-ray diffraction (HT-XRD) in a vacuum atmosphere was conducted from room temperature to 1000°C to observe in-situ the phase formation in the Ti_2AlN coating.

Following the production of Ti_2AlN MAX Phase, a series of oxidation tests are conducted to assess the coating's performance. These include isothermal oxidation for 10 hours at 850°C in laboratory air. Hereby, the Ti_2AlN MAX phase based coating develop a thermally grown layer of predominantly alumina, which is suitable as protection in high temperature environments. Below the alumina layer the Ti_2AlN MAX phase as well as the intermetallic Al-rich phase.

Analysis techniques included GD-OES for chemical composition, XRD for phase analysis, and SEM/EDS and TEM for structural evaluation are used.

10:40am **MA1-1-TuM-9 Empowering Pvd for Corrosion Protection: Ti(Al,Mg)Gdn Coatings with Game-Changing Corrosion Performance***, Holger Hoche* ***[holger_claus.hoche@tu-darmstadt.de]****,* Grafenstraße 2, Germany

Today, PVD technology is not the first choice for surface functionalization under corrosive conditions. The state-of-the-art for corrosion protection involves multilayers of electroplating or chemical corrosion protection layers, followed by a PVD top layer. This negatively affects sustainability and economic factors.

The authors successfully developed PVD-TiMgGdN and TiAlMgGdN coatings, sputtered with powder metallurgical targets in an industrial DC-magnetron PVD unit. With only a 5 µm coating thickness, corrosive mild steel substrates can be protected for at least 1000 hours in the salt spray test against corrosion [1]. By partially substituting magnesium with aluminum, the corrosion properties and manufacturability were further improved. Additionally, TiAlMgGdN coatings exhibit excellent corrosion behavior in alkaline (pH 8.5) and acidic (pH 5) environments.

This improvement is based on the synergistic effects of magnesium and gadolinium: Magnesium lowers the free corrosion potential of the coating, thereby reducing the susceptibility to galvanic corrosion. Gadolinium enhances the hydrophobicity of the surface, affects the conductivity, and supports the formation of stable passivation layers [2].

The influence of Gd, Mg, and Al on the corrosion protection performance will be investigated. Therefore, coatings with different Al/Mg proportions and varying Gd content were produced and compared regarding their microstructural, chemical, physical, and corrosion properties. Corrosion properties were investigated using different corrosion tests. The coating surfaces were also analyzed by nanoindentation measurements and chemical analysis to gather knowledge about the coating stability during corrosive stress.

The key properties influencing the corrosion protection effect of Ti(Al, Mg)Gd will be evaluated. Additionally, the effect of the specific chemical composition on the coating properties will be investigated. Understanding the key properties and their correlation with chemical composition allows for the specific design of functional corrosion-resistant PVD coatings.

[1] T. Ulrich, C. Pusch, H. Hoche, P. Polcik, M. Oechsner, Surface and Coatings Technology 422 (2021) 127496.

[2] H. Hoche, T. Ulrich, P. Kaestner, M. Oechsner, Vakuum in Forschung und Praxis 36, 2 (2024) 40.

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

Hard and Nanostructured Coatings I

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

8:00am **MA3-1-TuM-1 Hard TiAlTaN Coating by HIPIMS Deposition for Cutting Tools: Experiments, Simulations and Cutting Tests***, Emile Haye [emile.haye@unamur.be],* University of Namur, Belgium*; Jérôme Muller, Pavel Moskovin,* University of Namur, Innovative Coating Solutions, Belgium*; Loris Chavee,* University of Namur, Belgium*; Szilard Kolozsvari,* Plansee Composite Materials GmbH, Germany*; Stéphane Lucas,* University of Namur, Innovative Coating Solutions, Belgium **INVITED**

The quaternary $Ti_{1-x-y}Al_xTa_yN$ system has been shown to possess superior thin film properties compared to $Ti_{1-x}Al_xN$. In addition to the impact of Ta content, the sputtering technique significantly influences the structural and mechanical characteristics of these films. In this study, high power impulse magnetron sputtering (HiPIMS) was employed to deposit dense, tough, and hard $Ti_{1-x-y}Al_xTa_yN$ thin films from composite targets, which were then compared to TiAlN thin films. The effects of Ta addition were explored both experimentally and through simulations. VirtualCoater® was used to simulate thin film growth and properties, providing insight into the role of Ta in the densification process and the relationship between target composition and film composition. Subsequently, the mechanical, structural, and thermal properties of the films were experimentally examined, highlighting the significant benefits of Ta addition.

The observed enhancements are attributed to: (1) increased hardness due to film densification facilitated by intense Ta bombardment, (2) stabilization of the cubic structure at elevated temperatures, and (3) superior thermal resistance resulting from the formation of a mixed ($Ti_xAl_yTa_z$) oxide monolayer, as opposed to the Al_2O_3/TiO_2 bilayer observed in TiAlN-based coatings, as confirmed by XPS depth profiling.

Finally, dry cutting tool tests demonstrated a substantial increase in tool life and improved surface finish of the machined parts.

8:40am **MA3-1-TuM-3 Development and Comparison of AlTiN-Based HiPIMS Coatings for Microtool Machining Applications***, Ivan Fernández-Martínez [ivan.fernandez@nano4energy.eu],* Nano4Energy S.I.N.E, Spain

Currently, the coating of microtools plays a critical role in precision manufacturing, as tool performance and longevity are heavily influenced by the properties and quality of the coating employed. In this context, HiPIMS technology provides hard coatings with a smooth finishing, a low defect density, and homogeneous coverage of 3D intricate parts – an essential advantage when coating tools with diameters smaller than a millimeter – thus representing an ideal choice for these applications.

AlTiN-based coatings doped with silicon (AlTiSiN) and boron (AlTiBN) have been developed to meet the specific demands of micro-tool applications, such as enhanced wear resistance, high thermal stability, and low friction in extreme operating environments. Properties such as hardness, adhesion, and residual stress were tailored and correlated to HiPIMS process parameters. In addition to mechanical properties analysis, tool performance was evaluated through machining tests, selecting Hardened Steel (HRC60) and Ti6AlV4 alloy as case materials. Both the finishing of the machined parts, and the wear suffered by the tool were analyzed.

The results highlight the potential of HiPIMS-deposited AlTiN-based coatings to significantly extend tool life and improve machining quality in precision manufacturing. Furthermore, this study provides insights into the trade-offs between boron and silicon doping, offering practical guidelines for selecting the most appropriate coating for specific micro-tool applications.

Our findings underline the versatility of HiPIMS technology in tailoring thin-film properties for high-performance applications, demonstrating its growing relevance in the field of advanced microtools.

Keywords: hard coatings, nitrides, sputtering, HiPIMS.

9:00am **MA3-1-TuM-4 Micro-Fracture Toughness and Durability of HiPIMS-Deposited Hard Coatings used for Micro-Machining of $TiAl_6V_4$ Alloys***, Arley Garcia [arley.garcia@imdea.org],* Nano4Energy SL, IMDEA Materiales, Spain*; Jose Antonio Santiago,* Nano4Energy SL, Spain*; Christoph Kirchlechner,* Karlsruhe Institute of Technology (KIT), Institute for Applied Materials (IAM), Germany*; Pablo Diaz Rodriguez,* Nano4Energy SL, Spain*; Miguel Monclús,* IMDEA Materiales, Spain*; Iván Fernández Martínez,* Nano4Energy SL, Spain*; Alvaro Guzmán,* Universidad Politécnica de Madrid, Spain*; Subin Lee,* Karlsruhe Institute of Technology (KIT), Institute for Applied Materials (IAM), Germany*; Jon Molina Aldareguia,* Universidad Politécnica de Madrid, Spain

The High-Power Impulse Magnetron Sputtering (HiPIMS) technique enables the deposition of coatings with high hardness, low defect densities, and uniform, conformal coverage over complex 3D geometries, meeting the strict tolerances required in micromachining applications. High-speed machining (HSM) applications demand not only high hardness but also sufficient fracture toughness K_{IC}, which is critical for maintaining the structural integrity of both bulk and coated engineering components [1]. However, these mechanical properties are often antagonistic, particularly in materials capable of plastic deformation, where high hardness typically correlates with lower fracture toughness [2].

This study aims to systematically evaluate the fracture toughness and machining performance of AlTiN- and TiN-based coatings doped with Si, deposited by HiPIMS using different process parameters. Micro-fracture toughness was assessed on freestanding films using single cantilever bending tests, effectively minimizing residual stress and substrate interactions to obtain precise K_{IC} values. Crack formation at the cutting edge of a 0.4 mm diameter microdrill was observed using FIB, while the composition of the coatings was determined by GDOES. Additionally, XRD was employed to analyze grain growth texture and peak shifts, enabling the evaluation of biaxial stresses. The AlTiSiN coatings exhibited a hardness of 35 GPa, while TiSiN coatings reached 40 GPa. Fracture toughness ranged from 1.78 $MPa\cdot\sqrt{m}$ to 2.2 $MPa\cdot\sqrt{m}$, depending on the HiPIMS parameters used. In micromachining tests on the TiAl6V4 alloy, the coatings allowed continuous micro-milling to be extended from 40 minutes to over one hour. These findings link toughness values, stress reduction, and crack formation at the cutting edges with tool durability in machining applications.

[1]. BARTOSIK, M., et al. Fracture toughness of Ti-Si-N thin films. *International Journal of Refractory Metals and Hard Materials*, 2018, vol. 72, p. 78-82.

[2]. HAHN, Rainer, et al. Superlattice effect for enhanced fracture toughness of hard coatings. *Scripta Materialia*, 2016, vol. 124, p. 67-70.

9:20am **MA3-1-TuM-5 The Effects of Composition and Coating Thickness on the Mechanical Properties of TiZrN Coatings*, Chun Lin Yang [a0903271975@gmail.com], Jia-Hong Huang,*** National Tsing Hua University, Taiwan

The objective of this study is to deposit $Ti_{1-x}Zr_xN$ hard coatings on silicon substrates using an unbalanced magnetron co-sputtering (UBMS) technique and to investigate the effects of target current and deposition time on the structure and mechanical properties of the films. In the experiments, the Zr target current was adjusted from 0.17 A to 0.35 A, while the Ti target current was varied from 0.37 A to 0.19 A. The total target current was controlled at 0.54 A, resulting in three series of $Ti_{1-x}Zr_xN$ coatings, where x = 0.25, 0.55, and 0.85 (represented as Zr25, Zr55, and Zr85, respectively). The deposition time was also varied under these three compositional conditions to control the coating thickness, allowing the influence of composition and thickness on coating performance to be examined. The Ti and Zr targets were mounted on 2-inch unbalanced magnetron sputtering guns. The $Ti_{1-x}Zr_xN$ coatings displayed a strong (111) texture, with a texture coefficient exceeding 0.95. The results revealed that due to the solid solution strengthening mechanism, $Ti_{1-x}Zr_xN$ thin films exhibited high hardness across all three compositions, with the highest hardness of 34.9 GPa observed at Zr55. Furthermore, as the Zr content increased, residual stress also increased, attributed to the larger atomic radius of Zr compared to Ti. However, residual stress decreased with increasing coating thickness. This study uses the internal energy induced cracking (IEIC) method to calculate the stored elastic energy at different depths of the coatings based on residual stress, the Young's modulus of the films, and thickness. This approach enables precise calculation of energy gradients at various depths, thereby elucidating the stress distribution within the coatings.

9:40am **MA3-1-TuM-6 Superstoichiometric (Al,Cr)N*x* Coatings with Superior Hardness, Fracture Toughness, and Wear Resistance*, Fedor F. Klimashin [fedor.klimashin@empa.ch],*** Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland*; Martin Učík,* PLATIT a.s., Czechia*; Martin Matas, David Holec,* Montanuniversität Leoben, Austria*; Martin Beutner,* Otto-von-Guericke-Universität Magdeburg, Germany*; Jan Klusoň, Mojmír Jílek,* PLATIT a.s., Czechia*; Andreas Lümkemann,* PLATIT AG, Switzerland*; Johann Michler, Thomas E. J. Edwards,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland

Many transition-metal carbides, nitrides, and oxides are inherently non-stoichiometric compounds, characterised by broad homogeneity ranges in their phase diagrams. Deviations from stoichiometry, defined as the ratio of non-metal to metal atoms (x), can drastically affect properties. While substoichiometric compounds (x<1) have been widely studied, superstoichiometric compounds (x>1) remain largely unexplored.

Here we present our finding on superstoichiometric $(Al,Cr)N_x$ coatings sputter-deposited from an $Al_{60}Cr_{40}$ target at power densities reaching 840 W/cm^2. Experimental and computational analyses reveal that excess nitrogen predominantly occupies interstitial lattice sites. Upon surpassing a critical concentration ($x\approx1.06$), grain renucleation rates increase, disrupting columnar growth and altering the preferential orientation from (111) to (220). The coatings exhibit a single-phase, face-centred cubic structure, a dense microstructure, and reduced surface roughness compared to benchmark coatings produced by cathodic arc evaporation.

Remarkably, hardness, fracture toughness, and wear resistance equal or exceed those of the benchmark coatings. Our findings highlight the advantages of superstoichiometric $(Al,Cr)N_x$ as effective wear-resistant materials for advanced engineering applications, while also suggesting broader implications for the utilisation of superstoichiometric nitrides across various industries.

10:00am **MA3-1-TuM-7 Connecting Phase Stability and Mechanical Properties of Ti–-B–N Thin Films*, Rebecca Janknecht [rebecca.janknecht@empa.ch],*** Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland*; Tomasz Wójcik,* TU Wien, Austria*; Fedor F. Klimashin,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland*; Johann Michler,* EMPA, Switzerland*; Paul H. Mayrhofer, Rainer Hahn,* TU Wien, Austria

Understanding the relationship between thermally induced decomposition in metastable material systems and their mechanical properties is critical for designing thin films with improved wear resistance and thermal stability. Our previous work revealed that achieving improved solubility of B in fcc-TiN requires a deviation from the TiN-TiB tie line toward Ti-rich compounds, facilitated by the formation of vacancies in the non-metal sublattice. This deviation was achieved by non-reactive co-sputtering of a Ti target alongside TiN and TiB_2 targets, allowing full incorporation of 8.9 at.% B into the fcc-TiN lattice. In contrast, co-sputtering TiN and TiB_2 yielded compositions along the TiN-TiB_2 tie line, with excess B forming amorphous grain boundary phases [1]. In this study, we systematically annealed (1) a single-phase fcc-Ti–B–N coating with a composition of $Ti_{1.08}B_{0.18}N_{0.74}$ and (2) a Ti–B–N coating with amorphous B-rich grain boundary phases with a $Ti_{1.01}B_{0.21}N_{0.78}$ stoichiometry. Annealing was performed at 700°C, 800°C, 900°C, 1000°C, 1200°C and 1400°C for 10 minutes. Both coatings retained high hardness of 30±1 GPa up to 1200°C. The results of micro-cantilever bending tests indicate an inverse relationship between fracture toughness (K_{IC}) and annealing temperature (T_a). The $Ti_{1.01}B_{0.21}N_{0.78}$ coating exhibits a K_{IC} of 2.1 ± 0.1 $MPa\cdot m^{0.5}$, which increases to 3.8 ± 0.3 $MPa\cdot m^{0.5}$ upon increasing T_a to 1000°C, but K_{IC} for the $Ti_{1.08}B_{0.18}N_{0.74}$ sample was observed to decrease from 2.7 ± 0.1 $MPa\cdot m^{0.5}$ in the as-deposited state to 2.3 ± 0.1 $MPa\cdot m^{0.5}$ at T_a=1000°C. X-ray diffraction (XRD) and transmission electron microscopy (TEM) analyses confirm that K_{IC} improves only when metastable Ti–B–N decomposes into co-existing thermodynamically more stable fcc-TiN and hcp-TiB_2 phases without the formation of additional hcp-Ti precipitates. These findings highlight the critical influence of compositional and structural control on the thermal and mechanical stability of Ti–B–N thin films, providing new pathways for their use in high-performance applications.

[1] R. Janknecht et al., *A strategy to enhance the B-solubility and mechanical properties of Ti–B–N thin films*, Acta Mater., 271 (2024), Article 119858, 10.1016/j.actamat.2024.119858 [https://doi.org/10.1016/j.actamat.2024.119858]

10:20am **MA3-1-TuM-8 Effect of Oxygen Content and Thickness on the Property and Structure of Zr(O,N) Thin Film*, Chi Feng Hung [rbisme@gapp.nthu.edu.tw], Jia Hong Huang,*** National Tsing Hua University, Taiwan

Transition metal nitrides (TMeNs) have been widely applied as the protective coatings for tools due to their excellent properties. Zirconium nitride (ZrN) coatings, in particular, attract attention for the outstanding mechanical properties, corrosion resistance, and attractive golden color. It has been extensively reported that by adding oxygen in ZrN, the coating becomes Zr(N,O), where the ionic/covalent bond ratios can be tuned by adjusting the nitrogen-to-oxygen (N/O) ratio, and consequently influencing the optical, electrical, and mechanical properties of the coatings. However, most studies of Zr(N,O) coatings are on the effect of phase transition on structure and properties within a wide range of N/O contents, while limited research was conducted with range of oxygen content where Zr(N,O) remains a single phase. The purpose of this study is to explore the effect of oxygen content and film thickness on the structure and properties of single-phase Zr(N,O) films. In this study, the Zr(N,O) coatings were prepared using dc unbalanced magnetron sputtering with different durations and oxygen flow rates. Four different oxygen flow rates were used to control the oxygen content, and the coating thickness was controlled by varying the deposition times. The preferred orientation of the coatings was observed by X-ray diffraction. The results showed that dominant (111) and (200) textures appeared in the specimens with low and medium oxygen contents, respectively; in contrast, the texture of the specimens with high oxygen contents varied from random to (200) with increasing thickness. The hardness and Young's modulus of the coatings were nearly constant for all samples, with no observable trends with respect to thickness or oxygen content. The results revealed that the electrical resistivity increased with increasing oxygen content. The variation of residual stress with increasing oxygen content could be divided into two regimes, where compressive stress dropped sharply when the texture changed from (111) to (200) and then gradually decreased with further increase of oxygen content. The specimens with higher oxygen contents exhibited a significant decrease in electrical resistivity with decreasing thickness, while the specimens with medium oxygen contents showed a less distinct decrease, and the specimens with the lowest oxygen contents showed no change in resistivity with thickness. The residual stress also showed two trends, in which residual stress decreased with thickness for the specimens with the lowest oxygen contents, while residual stress increased with thickness for the other specimens.

10:40am **MA3-1-TuM-9 Effect of Fluence on Zirconium Nitride Coating Irradiated by 5-MeV-Proton*, Rou-Syuan Chen [250201chen@gmail.com],*** Department of Engineering and System Science, National Tsing Hua University, Taiwan (ROC)*; Kuan-Che Lan,* Institute of Nuclear Engineering and Science, National Tsing Hua University, Taiwan (ROC)

The development of advanced nuclear reactors and small modular reactors (SMR) will require nuclear fuel at a higher enrichment, which can introduce higher fluence of ionizing radiation such as fission products, neutron and proton etc. It will bring a severe challenge for fuel cladding materials. Zirconium nitride (ZrN) thin films are known for their thermal stability, high hardness, low resistivity, and better tribological properties, and they are widely used as hard coatings on deposited on the edge surface of cutting. Previous studies have also shown that ZrN exhibits great resistance to ionizing radiation. However, report about the irradiation damage of ZrN thin film as a function of proton fluence are rare. The objective of this study is to investigate on effect of fluence on ZrN thin film irradiated by 5-MeV-proton. Post irradiation examination of ZrN thin film will be performed to analyze the electronic properties, crystal structure, hardness and young' s modulus. The crystal structure and grain size of each sample are characterized by X-ray diffraction (XRD). The electronic resistivity is measured by a four-point probe. The film thickness is examined by scanning electron microscope (SEM). The residual stress is assessed by the laser curvature method (LCM) and average X-ray strain (AXS) method. The surface hardness and young' s modulus are measured by nanoindentation (NIP).

Protective and High-temperature Coatings
Room Town & Country A - Session MA1-2-TuA

Coatings to Resist High-temperature Oxidation, Corrosion, and Fouling II

Moderator: Vladislav Kolarik, Fraunhofer Institute for Chemical Technology ICT, Germany

2:20pm **MA1-2-TuA-3 Advanced Chemical Vapor Deposition Technology for High Temperature Applications***, **Natasa Djordjevic [natasa.djordjevic@ihi-bernex.com]**, Anne Zhang, Hristo Strakov,* IHI Bernex AG, Switzerland

Recent research explores the potential of Chemical Vapor Infiltration (CVI) and Chemical Vapor Aluminizing (CVA) technologies to produce advanced coating solutions for high temperature applications and materials with enhanced performance at demanding conditions.

CVI is increasingly used for establishing coating solutions for fiber-reinforced composites, enabling deposition of interface layers or infiltration of ceramic matrices with different precursors at elevated temperatures. The technique allows production of materials with greatly enhanced properties such as thermal stability, mechanical strength, oxidation and corrosion resistance.

On the other hand, CVA is a modern advanced process for applying diffusion coatings on metallic-based turbine blades and vanes in the hot section of aero- and land-based turbines against oxidation and corrosion. The CVA process is capable of controlled alloying the coating with additional elements by using metal chlorides and tight control of the coating composition and on this way increasing the life time of such components.

This work will highlight the latest developments of different coating technology solutions for high temperature applications, including improvements in precursor chemistry, reaction kinetics, stoichiometry and process control. Emphasis will be placed on the challenges related to maintaining uniformity and quality of deposition in different geometries and the influence of the coating equipment in order to precise control the parameters.

2:40pm **MA1-2-TuA-4 Oxygen Concentration Governs High-Temperature Oxidation Behavior of ($Cr_{0.5}Al_{0.5}$)(O_YN_{1-Y}) Thin Films***, **Pauline Kümmerl [kuemmerl@mch.rwth-aachen.de]**, Felix Leinenbach, Janani Ramesh,* RWTH Aachen University, Germany*; Daniel Primetzhofer,* Uppsala University, Sweden*; Marcus Hans, Jochen M. Schneider,* RWTH Aachen University, Germany

In (TM,Al)(O,N) (TM = Ti, V) thin films, the addition of oxygen enhances the thermal stability as for the decomposition into the hexagonal and cubic phases mobility on the metal and nonmetal sublattices is required, while for (TM,Al)N decomposition the activation of diffusion on the metal sublattice is sufficient. Little is known about the oxidation resistance of (TM,Al)(O,N) thin films; thus a systematic study of the influence of the O concentration in (Cr,Al)(O,N) on the oxidation resistance and oxide scale formation is presented here.

($Cr_{0.5}Al_{0.5}$)(O_yN_{1-y}) thin films were grown by reactive high power pulsed magnetron sputtering where the O content was systematically varied through adjustment of the O_2 partial pressure leading to compositions of $(Cr_{0.50}Al_{0.50})_{0.49}N_{0.51}$, $(Cr_{0.48}Al_{0.52})_{0.48}(O_{0.15}N_{0.85})_{0.52}$, and $(Cr_{0.44}Al_{0.56})_{0.46}(O_{0.40}N_{0.60})_{0.54}$. The oxidation behavior was investigated as a function of the O concentration at 1000 °C, 1100 °C, and 1200 °C for up to 16 h.

During oxidation an Al-rich oxide scale is formed. Between the ($Cr_{0.5}Al_{0.5}$)(O_yN_{1-y}) thin films and the scale, the formation of a an Al-depleted and O-enriched region is observed whereby the geometric extent and the level of porosity were strongly time and temperature dependent. At 1100 °C after 16 hours of oxidation the oxide scale thickness on $(Cr_{0.48}Al_{0.52})_{0.48}(O_{0.15}N_{0.85})_{0.52}$ was with 369± 48 nm significantly smaller than the 513± 96 nm and 462± 53 nm thick scale layers measured on $(Cr_{0.50}Al_{0.50})_{0.49}N_{0.51}$ and $(Cr_{0.44}Al_{0.56})_{0.46}(O_{0.40}N_{0.60})_{0.54}$, respectively. Furthermore, chemical environment dependent DFT calculations are performed to determine the species specific energy requirements for vacancy formation and mass transport in an effort to elucidate the time and temperature dependent oxidation behavior.

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

Thermal and Environmental Barrier Coatings I

Moderators: Sabine Faulhaber, University of California, San Diego, USA, **Fernando Pedraza**, La Rochelle University, Laboratory LaSIE, France, **Francisco Javier Perez Trujillo**, Universidad Complutense de Madrid, Spain, **Gustavo García-Martín**, REP-Energy Solutions, Spain

4:00pm **MA2-1-TuA-8 Multicomponent Rare Earth Oxide Coatings for Refractory Alloys***, Rachel Rosner, Kristyn Ardrey, Will Riffe, Alejandro Salanova, Prasanna Balachandran, Bi-Cheng Zhou, Carolina Tallon, Jonathan Laurer, Jon Ihlefeld, Patrick Hopkins, Sandamal Witharamage, **Elizabeth Opila [opila@virginia.edu]**,* University of Virginia, USA **INVITED**

Rare earth oxide (RE_2O_3) exhibit three crystal structures across the lanthanide series:hexagonal, monoclinic, and cubic, with all showing exceptionally high-melting temperatures (>2100°C) and excellent thermochemical stability.The cubic RE_2O_3, dysprosium through lutetium oxides, have isotropic thermal expansion with a reasonable match to Nb, making them suitable high temperature coatings for oxidation-prone refractory alloys.Multicomponent rare-earth oxides (MRO) allow the additional ability to target and optimize thermal expansion, resistance to molten deposits, and especially thermal conductivity, enabling their use as thermal/environmental barrier coatings (T/EBCs) in high-temperature, reactive environments such as turbine engines.Thermal conductivity of MROs has been shown to decrease with mixtures of RE_2O_3 with increasing mass and size variation.The larger, lighter, non-cubic lanthanide oxides, lanthanum through terbium oxides, mixed in a majority MRO cubic phase in non-equimolar proportions will precipitate as second phases once their solubility limit in the cubic RE_2O_3 is exceeded, enabling further reductions in thermal conductivity.In this work, MRO compositions are systematically varied to aid in achieving targeted thermal conductivity, thermal expansion, and resistance to molten deposits.Powder mixtures were combined, ball milled, and sintered via spark plasma sintering.Room temperature thermal conductivity was measured using the laser-based time domain thermoreflectance method.Thermal expansion was determined by dilatometry or lattice parameter measurements as a function of temperature.Resistance to molten CaO-MgO-Al_2O_3-SiO_2 was quantified after exposure at temperatures of 1300-1500°C for times between 1 and 96h.Here we evaluate whether a single layer MRO will meet all design requirements for a (T/EBC) enabling cost efficient coating synthesis or whether additional coating layers are required to achieve adherent, protective properties for hot section turbine engine component applications.

4:40pm **MA2-1-TuA-10 PVD Ce-Coating to Mitigate Intergranular Oxidation of Additively Manufactured Ni-Base Alloy In625***, **Anton Chyrkin [chyrkin@chalmers.se]**, Andrea Fazi, Mohammad Sattari, Mattias Thuvander,* Chalmers University of Technology, Gothenburg, Sweden*; Wojciech J. Nowak,* Rzeszow University of Technology, Rzeszow, Poland*; Dmitry Naumenko,* Forschungszentrum Jülich GmbH, Germany*; Jan Froitzheim,* Chalmers University of Technology, Gothenburg, Sweden

Additively manufactured (AM) Powder Bed Fusion - Laser Beam Ni-base alloy IN625 suffers from intergranular oxidation (IGO) during air exposure at 900 °C in contrast to the conventionally manufactured (CM) forged alloy IN625. A new mechanism of IGO in AM alloys is proposed: IGO is triggered by oxide buckling over the grain boundaries (GBs) in the alloy followed by oxidation of the open intergranular voids. Application of a 10 nm thick PVD coating of Ce promoted a slower inward growth of the Cr_2O_3 scale, better oxide adherence and as a result strongly suppressed IGO. The Cr_2O_3 scales thermally grown on both uncoated and Ce-coated alloys were analysed with SEM/EDX, EBSD, GD-OES, TEM and APT. The main beneficial effect provided by the Ce-coating is improved oxide adherence preventing oxide from buckling and oxidation of intergranular voids.

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

Hard and Nanostructured Coatings II

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

1:40pm **MA3-2-TuA-1 Designing Nanocrystalline Alloys and Compounds: Unraveling Compositional and Microstructural Pathways to Exceptional Properties***, **Rostislav Daniel [rostislav.daniel@unileoben.ac.at]**, Michal Zitek, Tobias Ziegelwanger,* Montanuniversität Leoben, Austria*; Ranming Niu,* The University of Sydney, Australia*; Edoardo Rossi, Marco Sebastiani,* Università degli studi Roma Tre, Italy*; Petr Zeman, Stanislav Haviar,* University of West Bohemia, NTIS, Czechia*; Jozef Keckes,* Montanuniversität Leoben, Austria **INVITED**

This talk presents advanced methods in combinatorial synthesis and microstructural design to achieve extraordinary properties in multielement alloys and layered coatings. Using the CrCuTiW alloy system as a primary example, we demonstrate how large compositional variations and the limited miscibility between elements lead to diverse self-assembled multicomponent phases,combining solid solutions, nanocomposites, and metallic glasses. These structures exhibit unexpected combinations of hardness and elastic modulus, demonstrating the potential for unique property tailoring.

In a second example, a cross-sectional combinatorial synthesis of nanostructured CrMnFeCoNi alloy is employed to address the thermal stability of this metastable alloy. This approach enables an in-depth analysis of segregation kinetics in the primary phase at moderate temperatures (50-450°C) resulting in the formation of a variety of coexisting phases that enhance alloy strength while maintaining ductility and fracture toughness. This approach demonstrates its capability to provide insights into the thermal behavior of complex, metastable microstructures and allows for controlled property enhancement.

Additionally, the talk emphasizes a bio-inspired approach to compositional and microstructural design withina layered Zr-Cu-N system, where antibacterial properties are combined with enhanced fracture toughness and stress resistance. These multifunctional coatings represent a new class of sustainable materials, suitable for both hard and smart coating applications.

Our methodology integrates advanced multi-technique characterization tools, including 2D (XRD, EDX) and 3D (nano-XRD, nanoindentation) mapping capabilities, combined with transmission electron microscopy and atom probe tomography. These techniques facilitate a rapid assessment of processing-structure-property relationships in these novel nanostructured alloys, bridging the gap between theoretical predictions and practical applications. Together, these methodologies provide a pathway to the design of next-generation multifunctional layered architectures,tailored down to the nanoscale, to enable exceptional mechanical and functional properties and robust thermal stability.

2:20pm **MA3-2-TuA-3 Evolution of the Pulsed-DC Powder-Pack Boriding Process: Exploring Low-Temperature Boride Layer Formation***, **J.L. Rosales-Lopez [jrosalesl1401@alumno.ipn.mx]**[1], M. Olivares-Luna, L.E. Castillo-Vela, I.E. Campos-Silva,* Instituto Politécnico Nacional, Mexico

This study rigorously investigates the transformative potential of the Pulsed-DC Powder-Pack Boriding (PDCPB) process to catalyze boride layer formation on AISI H13 steel at remarkably reduced temperatures (600°C, 650°C, and 700°C) under substantial current densities (~952mA·cm^{-2}) and significantly minimized exposure times of 1800s, 2700s, and 3600s. Enabled by the implementation of a custom high-capacity power supply, this innovation generates the essential electric field to support boriding at unprecedented low temperatures. Traditionally, achieving similar results in AISI H13 required treatments at temperatures exceeding 900°C with exposure times of at least 14400s, underscoring the extraordinary advancement represented by this approach.

Through meticulous microstructural and physicochemical analyses using SEM–EDS and XRD, the study reveals substantial findings: at a mere 600°C, PDCPB successfully produced dense, biphasic FeB+Fe_2B layers with thicknesses ranging from ~8μm to ~17μm, uniformly distributed across the sample surfaces. Remarkably, and contrary to established reports on borided AISI H13, the substrate retained its α-phase microstructure without transformation to the α'-phase, and the interface between the boride layer and substrate remained free of any diffusion zone. This breakthrough not only introduces significant commercial scalability for low-temperature boriding but also opens possibilities for further innovations, potentially achieving effective boriding near the 530°C threshold. The insights presented mark a seminal advancement in boriding technology, with vast implications for industrial applications and the future of materials engineering.

2:40pm **MA3-2-TuA-4 Three-Fold Superstructured HfN/HfAlN Multilayers***, **Marcus Lorentzon [marcus.lorentzon@liu.se]**[2],* Linköping University, IFM, Thin Film Physics Division, Sweden*; Rainer Hahn,* TU Wien, Institute of Materials Science and Technology, Austria*; Lars Hultman, Justinas Palisaitis,* Linköping University, IFM, Thin Film Physics Division, Sweden*; Johanna Rosen,* Linköping University, IFM, Materials Design Division, Sweden*; Grzegorz Greczynski, Jens Birch, Naureen Ghafoor,* Linköping University, IFM, Thin Film Physics Division, Sweden

Brittleness and poor fracture toughness are limiting factors for the application of hard protective coatings. To resolve these issues, we explore multilayer superlattice (SL) coating designs based on $HfN_{1.33}$ and $Hf_{0.76}Al_{0.24}N_{1.15}$. We achieve high-quality single-crystal films and superlattices with superior mechanical characteristics by epitaxial growth on MgO(001) substrates using ion-assisted reactive magnetron sputtering at high temperatures.

The structure and properties of monolithic single-crystal $HfN_{1.33}$ and $Hf_{0.76}Al_{0.24}N_{1.15}$ are studied to evaluate the SL-coating performance. Overstoichiometric HfN_y exhibits metal-like ductility in micropillar compression tests, with easy dislocation nucleation and movement along multiple {111}<110> slip systems, which results in significant strain hardening and a doubled ultimate strength at 17% strain, compared to the yield point at 2%. The improved ductility is attributed to point defects—vacancies and nitrogen interstitials—forming a checkerboard superstructure of hyper-overstoichiometric and near-pristine domains. In contrast, HfAlN shows improved hardness and yield strength in pillar compression, however, it fails by strain-burst with fractures on the {110}<110> slip system. These properties stem from strain fields, pinning dislocations, which develop between coherent Hf- and Al-rich nanodomains, formed by surface-initiated spinodal decomposition. In addition, the domains similarly self-organize into a checkerboard superstructure.

Thus, by combining overstoichiometric $HfN_{1.33}$ and $Hf_{0.76}Al_{0.24}N_{1.15}$ in SLdesigns with equal layer thicknesses but varying bilayer period of 20 nm, 10 nm, and 6 nm, fascinating three-fold superstructured SLs are created by checkerboard superstructuring in 1) the HfN layers and 2) the HfAlN layers, as well as 3) the multilayer structure itself. While the interfaces provide dislocation pinning to maintain an equally high hardness as $Hf_{0.76}Al_{0.24}N_{1.15}$, about 20% higher than $HfN_{1.33}$, other multilayer effects and inherent ductility of $HfN_{1.33}$ enhance the toughness through coherency strains, crack-tip blunting or deflection. The SLs are analyzed using X-ray diffraction, reciprocal space maps, high-resolution z-contrast scanning transmission electron microscopy, selected area electron diffraction, nanoindentation, and micropillar compression tests. Post-mortem imaging of the pillars reveals the underlying plastic deformation mechanisms. Superlattice effects enhance mechanical performance, combining properties of both materials for coatings with high hardness and improved toughness, ideal for advanced protective applications.

3:00pm **MA3-2-TuA-5 Effects of Different Interlayer Layers on Residual Stress Relief in γ-MoN/Ti and γ-MoN/Mo Thin Films***, **Ding-Hsuan Yang [dave35116@gmail.com]**, Jia-Hong Huang,* National Tsing Hua University, Taiwan

Transition metal nitrides have been widely used due to their outstanding properties such as wear resistance, high corrosion resistance and excellent mechanical properties. γ-Mo_2N coating is becoming more popular in terms of high temperature tribological properties, which results from the formation of Magnéli oxide phase. However, residual stress from the deposition of the hard coatings is a common issue that may decrease the adhesion strength and fracture toughness. Adding a metal interlayer is a convenient method to relief the residual stress of hard coatings. The purpose of this research was to compare the behavior of stress relief by using different metal interlayers, Ti and Mo. In this study, the Ti and Mo interlayers were deposited by DC-unbalanced magnetron sputtering, while the γ-Mo_2N coatings with Ti and Mo interlayer were deposited using high

[1] **Graduate Student Award Finalist**

[2] **Graduate Student Award Finalist**

pulsed power magnetron sputtering on Si (100) substrates. The γ-Mo_2N layer thickness was maintained at 1000 nm with three different interlayer thicknesses controlled at 50, 100, and 150 nm. The overall residual stress of the bilayer coatings was determined by the laser curvature method (σ_{LCM}), while individual layer stress was evaluated by the average X-ray strain method (σ_{AXS}). Contrary to our expectations, the results show that σ_{LCM} values are consistently higher than σ_{AXS}, suggesting that the Ti and Mo interlayer cannot effectively relieve stress through plastic deformation. The Ti interlayer may be partly converted to TiN due to the reaction of N_2 gas or N_2^+ ions with the pre-deposited Ti, and consequently the interlayer cannot be plastic deformed to relieve stress. In contrast, due to the high elastic constant of Mo, the compressive residual stresses in the Mo interlayer is higher than that in γ-Mo_2N coating, where the stress is higher than the yield strength of Mo metal, indicating that Mo interlayer cannot serve as a buffer layer to relieve residual stress of γ-Mo_2N coatings.

4:00pm **MA3-2-TuA-8 Highly Stacked Ge0.8Si0.2 Nanosheets Fabricated by Wet Etching of MBE-Grown Superlattice Films***, Zefu Zhao, Dun-Bao Ruan,* FZU-Jinjiang Joint Institute of Microelectronics, College of Physics and Information Engineering, School of Advanced Manufacturing, Fuzhou University, China*; Kai-Jhih Gan,* FZU-Jinjiang Joint Institute of Microelectronics, College of Physics and Information Engineering, School of Advanced Manufacturing, Fuzhou University,, China*; **Qian Cheng Yang [455783022@qq.com]**,* FZU-Jinjiang Joint Institute of Microelectronics, College of Physics and Information Engineering, School of Advanced Manufacturing, Fuzhou University, China*; Kuei-Shu Chang-Liao,* Department of Engineering and System Science, National Tsing Hua University, Taiwan*; Jie-yin Zhang,* SongShan Lake Materials Laboratory, Center for Semiconductor Heterogeneous Materials and Devices, Dongguan 523830, China*; Shenglin Pan,* FZU-Jinjiang Joint Institute of Microelectronics, College of Physics and Information Engineering, School of Advanced Manufacturing, Fuzhou University, China

This work demonstrates a novel wet etching approach to fabricating a 3-stacked Ge0.8Si0.2 nanosheet structure, which holds significant promise for advancing next-generation transistor technologies. As the semiconductor industry approaches the 2 nm node, transistors with nanosheet architectures are emerging as strong contenders to replace FinFETs due to their superior electrostatic control over short channels, enabling enhanced performance and power efficiency. In this study, high-quality and atomically flat superlattice Ge0.8Si0.2 layers were epitaxially grown using molecular beam epitaxy (MBE), providing an ideal platform for nanosheet fabrication. The key innovation lies in the utilization of HNO_3-based wet etching, which exploits the high selectivity of Ge over Ge0.8Si0.2 to achieve precise and uniform etching, resulting in three stacked Ge0.8Si0.2 nanosheets with exceptional channel uniformity. To mitigate lattice mismatch and confine misfit dislocations, an undoped Ge buffer layer was grown on a Si wafer, serving as a critical intermediate layer. Reciprocal space mapping (RSM) analysis confirms the successful realization of a fully relaxed Ge buffer layer and strained Ge0.8Si0.2 nanosheets, highlighting the structural integrity and strain engineering of the fabricated stack. This work not only provides a scalable and cost-effective method for nanosheet fabrication but also paves the way for the integration of GeSi-based nanosheet transistors in future sub-2 nm technology nodes, offering a pathway to further miniaturization and performance enhancement in semiconductor devices.

4:20pm **MA3-2-TuA-9 A T*EM* and Nanoindentation Study of the Correlation between Composition, Structure and Mechanical Properties of the AlCu Thin Film System***, **Dániel Olasz [olasz@student.elte.hu]**, Quang Chinh Nguyen,* Eötvös Loránd University, Hungary*; Noémi Szász, György Sáfrán,* HUN-REN Centre for Energy Research, Hungary

AlCu alloys in their bulk form are of great importance of the industry, including construction and aerospace and also their study has been instrumental in understanding the precipitation hardening effect in alloys. However, they are less frequently studied in their thin film form, with existing research mainly focusing on low alloying concentration cases. The aim of the present research is to investigate over the whole compositional range the correlation between composition, structure and mechanical properties in the AlCu thin film system.

By applying the micro-combinatorial approach, 15 discrete $Al_{1-x}Cu_x$ ($0 \le x \le 1$) films having a thickness of 1.7 um and different compositions were deposited on a single Si substrate through DC magnetron sputtering of Al and Cu in a single vacuum run. Nanoindentation measurements have demonstrated that even for pure Al and Cu, the layers exhibit significant strength in comparison to their bulk counterparts. The hardness increases substantially with increasing alloying concentration, reaching a maximum of about 16 GPa in the vicinity of ~50 at% Cu composition. For these layers, which display the highest strength, the loading stage of the indentation curves exhibits a step-like behavior, indicative of non-continuous deformation. Furthermore, the indentation experiments on the layer system provide an excellent opportunity to gain a deeper understanding of the indentation size effect (ISE), as the hardness of the layers at low alloying concentrations is independent of the applied maximum indentation force, whereas a clear sensitivity to ISE can be observed for layers containing 40-70 at% Cu. A comprehensive TEM study over the whole Al-Cu composition range using cross-sectional FIB TEM lamellae revealed microstructural features such as grain size, crystal structure, presence of non-equilibrium phases and layer growth mechanisms. This, together with the nanoindentation results, led to an understanding of the deformation mechanisms at work during indentation and hence to an explanation of the surprising mechanical properties of thin AlCu layers.

Protective and High-temperature Coatings
Room Town & Country D - Session MA4-1-TuA

High Entropy and Other Multi-principal-element Materials I

Moderators: Shih-Hsun Chen, National Yang Ming Chiao Tung University (NYCU), Taiwan , **Pavel Soucek**, Masaryk University, Czechia

1:40pm **MA4-1-TuA-1 Phase-Adjustable High-Entropy Alloy Coatings Prepared via Thermal Spray Process***, **Shih-Hsun Chen [brucechen@nycu.edu.tw]**,* NYCU, Taiwan **INVITED**

The alloy design in HEAs has impacts the resultant microstructure, phase structure, and hardness and wear resistance. Careful selection of HEAs elements is critical depending on the intended application of the alloy. Development of HEAs alloys follows careful selection of elements such a Co, Cr, Ni, Al, Fe, Ti among others. Mechanical properties in HEAs depend on existing phases whether single phase (BCC or FCC) or a mixture of phases and intermetallics or oxides. AlCoCrFeNi alloy has been widely studied and is considered an excellent base for additional strengthening strategies through compositional optimization. In this study, $Al_{0.5}CoCrFeNi_2Ti$ powders were prepared using the gas atomization method, and annealing treatments were performed to characterize the phase transformation behavior, providing essential insights into the effects of thermal energy during the atmospheric plasma spraying process. To further explore these effects, the study examined effects of plasma energy and powder size in the production of $Al_{0.5}CoCrFeNi_2Ti$ coatings. The characterization of both powders and coatings was consolidated to better understand the influence of Ti addition on the $Al_{0.5}CoCrFeNi_2$ HEA system and to assess the performance of its plasma-sprayed coatings.

2:20pm **MA4-1-TuA-3 Three Noble Metals, Three Different Stories: Unraveling the Complex Behavior of Cu, Ag, and Au in CrMnFeCoNi High-Entropy Alloy Thin Films***, **Salah-eddine Benrazzouq [salah-eddine.benrazzouq@univ-lorraine.fr]**,* Institut Jean Lamour - Université de Lorraine, France*; Ekaterina V. Gunina, Svyatoslav Povarov,* School of Physics and Engineering, ITMO University, Russian Federation*; Jaafar Ghanbaja, Sylvie Migot, Alexandre Nominé, Jean-François Pierson, Valentin A. Milichko,* Institut Jean Lamour - Université de Lorraine, France

Advanced electronic devices and sensing technologies demand materials with precisely tunable electrical and optical properties, alongside excellent structural stability. While high-entropy alloys (HEAs) show promise for such applications due to their unique multi-element composition, controlling their functional properties remains challenging. Noble metals (Cu, Ag, Au) were strategically chosen for this study due to their similar electronic configurations and increasing metallic radii (rCu=0.127 nm, rAg=0.144 nm, rAu=0.147 nm) compared to the average metallic radius of the Cantor alloy (0.125 nm).

This study unravels the distinct stories of how Cu, Ag, and Au additions transform the structure and properties of CrMnFeCoNi Cantor alloy thin films, revealing behaviors that challenge our initial expectations. Using DC magnetron co-sputtering, we systematically investigated these transformations through comprehensive characterization including X-ray diffraction (XRD), high-resolution transmission electron microscopy (HRTEM), electrical resistivity measurements, and non-linear optical response.

Each noble metal reveals a unique chapter in phase evolution and property modification, far from the expected systematic progression based on atomic size. Copper tells a story of structural preservation, maintaining the fcc structure while systematically reducing nano-twin density, culminating in near-zero TCR (-2.86 ppm/K) at 37 at% Cu. Silver writes a different narrative through unexpected phase separation, creating artistic patterns of nano-precipitates with a characteristic tweed-like microstructure, despite predictions of solid solution formation. Gold presents perhaps the most surprising tale, where HRTEM unveils large grains with intricate twin boundary networks, contrasting sharply with its apparent amorphous nature in XRD analysis and defying expectations based on atomic size considerations.

These distinct structural modifications yield equally diverse functional properties. Cu-modified films demonstrate precise control over electrical behavior while maintaining metallic characteristics, aligning with initial predictions. Ag-modified films combine decreased electrical resistivity with enhanced second harmonic-generation (SHG) response, providing unexpected opportunities for multifunctional properties. Au-modified films exhibit unique optical properties tied to their complex grain structure. These three different stories highlight how noble metals can orchestrate dramatically different transformations in HEA thin films, while comparing their properties with sustainability metrics offers guidance for future technological implementations

2:40pm **MA4-1-TuA-4 Mechanical, Tribological and Corrosion Behavior of CoCrFeNiMn High-Entropy Thin Films***, **Lin Wu [lin.wu2@mail.mcgill.ca]**,* McGill University, Canada*; León Zendejas Medina,* McGill University, KTH Royal Institute of Technology, Canada*; Richard Chromik, Janine Mauzeroll,* McGill University, Canada

The CoCrFeNiMn (Cantor alloy) thin films deposited under ambient and high temperature conditions have been studied from the aspect of microstructure, mechanical, tribological and corrosion properties. The Cantor films were deposited by pulsed direct current magnetron sputtering on silicon wafers at ambient and 350 °C. An FCC phase appeared in both ambient and high temperature films, with small amounts of an unidentified secondary phase. Using nanoindentation, film hardness (H) and reduced elastic modulus (Er) were measured.

Micro-tribology testing was conducted using a 20 μm radius spherical diamond tip in the dry air atmosphere (RH% value under 4.0%), applying normal loads ranging from 2 to 7 mN. The worn surfaces were characterized by atomic force microscopy. Schiffmann's model was used to evaluate the elastic and plastic components of the friction. The corrosion behaviors of the films were studied by using anodic polarization in 3.5% NaCl, followed by a tribological testing carried out on the corroded surfaces, to address the correlation of corrosion response with the phase composition, mechanical properties and tribological behavior of the films.

3:00pm **MA4-1-TuA-5 Microstructure and Mechanical Properties Evaluation of CoCrNiTiAl Multiple-Principal Element Alloy Thin Films: Effect of TiAl Additions***, **Pongpak Chiyasak [pongpak.c@ku.th]**,* Department of Materials Engineering, Faculty of Engineering, Kasetsart University, Thailand*; Jun-Xing Wang,* Ming Chi University of Technology, Taiwan*; Chia-Lin Li,* Center for Plasma and Thin Film Technologies, Ming Chi University of Technology, Taiwan*; Surapit Posri, Thanawat Santawee, Worawat Wattanathana, Aphichart Rodchanarowan,* Department of Materials Engineering, Faculty of Engineering, Kasetsart University, Thailand*; Jyh-Wei Lee,* Ming Chi University of Technology, Taiwan

Multiple-principal element alloy thin films have attracted lots of interest from academia due to their optimized properties for both functional and structural applications, for example, high strength, good thermal stability, cost efficiency, and lower stacking fault energy. Several previous studies have shown that adding Ti and Al as alloying elements into CoCrNiTiAl multiple-principal element alloy thin films could further enhance their performance. In general, Ti element can transform the face-centered cubic structure into the amorphous structure, while Al generates body-centered cubic (BCC) structures, promoting higher hardness and wear resistance. However, the effect of the addition of Ti and Al elements into CoCrNiTiAl multiple-principal element alloy thin films is still unexplored.

In this work, the CoCrNiTiAl multiple-principal element alloy thin films with different amounts of Ti and Al contents were fabricated by the co-sputtering of TiAl and CoCrNi targets through a hybrid magnetron sputtering system. The CoCrNi and TiAl target were connected with a high power impulse magnetron sputtering system and a radio frequency power supply, respectively. The power input of the TiAl target was adjusted to achieve multiple-principal element alloy thin films with different TiAl contents.The field-emission (FE) scanning electron microscopy, FE-electron probe microanalyzer, X-ray diffraction, nanoindentation, tribometer, and electrochemical workstation were used to characterize microstructure, chemical composition, phase evolution, hardness, wear resistance, and corrosion behavior of multiple-principal element alloy thin films. The effect of TiAl contents on the microstructure and mechanical properties of CoCrNiTiAl multiple-principal element alloy thin films were discussed in this work.

Keywords: multiple-principal element alloy, CoCrNiTiAl, phase transformation, mechanical properties

4:00pm **MA4-1-TuA-8 Effects of Deposition Parameters and Post-Annealing Treatment 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*; Sen-You Hou,* Department of Materials Science and Engineering, National Tsing Hua University, Taiwan*; Li-Chun Chang,* 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*; Po-Yu Chen,* Department of Materials Science and Engineering, National Tsing Hua University, Taiwan

TiZrNbTaMo high-entropy alloys (HEAs) with a body-centered cubicstructure are known for their excellent compressive yield strength and significant compressive plasticity, retained even in thin film forms. These properties make them promising for various applications.The deposition parameters significantly influence the density and microstructure of thin films, affecting mechanical properties. In this study, TiZrNbTaMo high-entropy alloy films (HEAFs) were prepared using high power impulse magnetron sputtering (HIPIMS), DC, and RF power sources. The effects of pulse frequency and duty cycle in HIPIMS on their structure and properties were systematically investigated.The microstructure and crystal structure of TiZrNbTaMo HEAFs were characterized using transmission electron microscopy (TEM) and X-ray diffractometry (XRD), while their mechanical properties were evaluated by nanoindentation.TiZrNbTaMo HEAFs deposited by HIPIMS exhibited increased hardness due to higher peak power density resulting in the coexistence of amorphous and nanocrystalline structures. However, the highest hardness of 5.78 GPa was achieved with RF power source, which was attributed to more nanocrystalline content and stacking faults.To further improve the mechanical properties of HEAs, post-annealing is used to modify the grain size and structure by inducing microstructures such as stacking faults and twins. In this study, the effects of post-annealing at 900 °C on microstructure and mechanical properties were investigated, and detailed features such as grain sizes, annealing twins, and variations in mechanical properties were discussed.

4:20pm **MA4-1-TuA-9 High Entropy Alloys Coatings for Inertial Confinement Fusion Hohlraums***, **Leonardus Bimo Bayu Aji [bayuaji1@llnl.gov]**,* *Daniel Goodelman, David Strozzi, Brandon Bocklund, Scott Peters, Alison Engwall, Swanee Shin, Gregory Taylor, Eunjeong Kim, James Merlo, Sergei Kucheyev,* Lawrence Livermore National Laboratory, USA

Hohlraums, centimeter-scale sphero-cylindrical heavy-metal canswith wall thickness of 10 - 100 μm, are a key component of an indirect-drive inertial confinement fusion (ICF) target as it determines the x-ray drive that implodes the fuel capsule. A previous study [Jones et al., Phys. Plasmas 14, 056311 (2007)] has demonstrated the feasibility of improving the x-ray drive by using a hohlraum made from a mixture ("cocktail") of elements, instead of a single element hohlraums, such as Au or U traditionally used for ICF. Here, we present our results on developing sputter-deposited heavy-metal high-entropy alloys (HEAs) for hohlraums with improved x-ray drive, high electrical resistivity to support a magnetized ICF, and material properties that are compatible with the ICF target fabrication process.

This work was performed under the auspices of the U.S. DOE by LLNL under Contract DE-AC52-07NA27344 and LDRD project 23-ERD-005.

4:40pm **MA4-1-TuA-10 Evaluation of the Microstructure and Electrocatalytic Performance of FeNiMoWCux High-entropy Alloy Thin Film***, **Kuan-Chen Lin [love17321125@gmail.com]**,* Ming Chi University of Technology, Taiwan*; Bih-Show Lou,* Chang Gung University, Taoyuan city, Taiwan., Taiwan*; Chia Lin Li, Naveen Karuppusamy, Thi Cam Tuyen, Jyh-Wei Lee,* Ming Chi University of Technology, Taiwan

Since Professor Yeh's introduction of high-entropy alloys (HEAs) in 2004, research on HEA materials has garnered worldwide attention due to their exceptional properties. Certain HEA materials have demonstrated great potential as electrocatalysts, attributed to their unique structures and compositions. In this study, $FeNiMoWCu_x$ HEA thin films with varying Cu contents were synthesized on Si wafers and nickel foams using magnetron sputtering. Co-sputtering was performed with equimolar FeNiMoW and pure Cu targets to produce $FeNiMoWCu_x$ films with x = 0, 0.5, and 1.0. An inclined deposition technique was employed to increase surface roughness and create more active sites on the HEA films. A comprehensive evaluation of the films was conducted, examining their composition, microstructure, crystallographic structure, surface roughness, adhesion, and corrosion resistance in 0.5 M sulfuric acid. Furthermore, electrochemical oxygen evolution reaction (OER) and hydrogen evolution reaction (HER) performance along with long term stability were carried out using a three-electrode electrochemical configuration in 1.0 M KOH and 1.0 M KOH + 3.5 wt.% NaCl solutions, respectively, to assess their electrocatalytic properties. The findings revealed that the $FeNiMoWCu_x$ HEA films hold considerable promise for electrode surface modification in water-splitting applications, owing to their improved catalytic activity, structural stability, and excellent corrosion resistance.

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

Thermal and Environmental Barrier Coatings II

Moderators: Fernando Pedraza, La Rochelle University, Laboratory LaSIE, France, **Francisco Javier Pérez Trujillo**, Universidad Complutense de Madrid, Spain

8:40am **MA2-2-WeM-3 Comparison of the Protective Performance of YSZ Coatings on Austenitic Steel Under Static and Dynamic Molten Carbonate Conditions***, M. Teresa de Miguel,* ***Gustavo García Martín [gusgarci@ucm.es]****, M. Isabel Lasanta, Jaime Chaves, Francisco Javier Perez Trujillo,* Universidad Complutense de Madrid, Spain*; Pauline Audigié, Sergio Rodríguez, Alina Agüero,* Instituto Nacional de Técnica Aeroespacial (INTA), Spain

The development of protective coatings is crucial for mitigating the severe corrosion caused by molten salts operating at high temperatures in Concentrating Solar Power plants. While nitrate-based salts are currently used as thermal storage media in CSP plants, carbonate salts offer superior thermal stability. The properties of the Li-Na-K carbonate eutectic were enhanced by the addition of 0.5% of α-Al_2O_3 nanoparticles. The new mixture presents a reduced melting point and an increase in the decomposition temperature, allowing the operation of the CSP plants at 700 °C. This increase would enhance steam generation efficiency and would reduce the Levelized Cost of Electricity (LCoE). However, the intense corrosive effects of molten carbonates demand the development of durable protective coatings to extend the lifespan of critical components.

This study evaluates the performance of yttria-stabilized zirconia (YSZ) sol-gel coatings applied to the 310H austenitic steel when it is exposed to static and dynamic conditions in a lab-scale setup. Corrosion tests were conducted at 700°C for up to 2500 hours in the eutectic ternary Li_2CO_3–Na_2CO_3–K_2CO_3 mixture with α-Al_2O_3 additive. The protective behavior of the coatings was assessed through gravimetric analysis, microstructural characterization and XRD. The performance of the coated material was also compared with the uncoated substrate. The coated samples exhibited an improved corrosion resistance, whereas the uncoated steels showed substantial degradation, including detachment and high mass variation. The presence of the YSZ coating reduced the corrosion extent by over one-third, with the thickness of the corrosion products measuring approximately 200 µm - 300 µm on the uncoated substrate, and between 50 µm - 70 µm on the coated samples.

The YSZ coating exhibited very similar behavior under both dynamic and static conditions. No significant difference was observed in the thickness of the corrosion product layer. Additionally, the same multilayer structure was identified, with the outer layer mainly composed of $LiFeO_2$, while the inner region was enriched in iron and chromium oxides. However, the uncoated austenitic steel showed higher degradation when exposed to dynamic conditions, displaying cracks along the corrosion layer and detachments.

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

9:00am **MA2-2-WeM-4 Enhanced Oxidation Resistance of Ni substrate by Sputtered Nanotwinned $Al_9SiCo_{20}Cr_{20}Ni_{45}NbMo_4$ Medium-Entropy Alloy Thin Films at High Temperatures***,* ***Jun-Hui Qiu [junhui-qiu@gapp.nthu.edu.tw]****, Yi-Chun Yen, Fan-Yi Ouyang,* Department of Engineering and System Science, National Tsing Hua University, Taiwan

High-entropy alloys exhibit various properties, such as superior thermal stability, oxidation resistance, and corrosion resistance. These characteristics have sparked interest in using HEAs as anti-oxidation protective coatings. The nanotwinned structure within these alloys contributes to their high hardness and thermal stability, while the sluggish diffusion in high entropy alloys helps lower oxidation rates.

In this study, $Al_9SiCo_{20}Cr_{20}Ni_{45}NbMo_4$ medium-entropy alloy thin films with a nanotwinned structure were successfully fabricated using magnetron sputtering system on a nickel-metal substrate. Then, the samples were subjected to high-temperature oxidation tests at 600°C, 700°C, and 800°C for 72 hours in dry air using a thermogravimetric analyzer to investigate the high-temperature oxidation behavior of these films and their protective effects against oxidation on nickel substrates. The results demonstrated that the medium-entropy alloy films exhibited strong oxidation resistance, leading to significantly lower oxidation rates and mass gain than pure nickel. The oxidized films had smoother surfaces than bare nickel substrates, with no pores or cracks. Due to grain growth at high temperatures, the (111) texture and nanotwinned structure in the films partially disappeared at 600°C and 700°C, although some twin structures remained. At 800°C, the twin structures were nearly absent, forming larger grains and a more pronounced (200) diffraction peak. After 24 hours of oxidation at 800°C, chromium oxide particles began to precipitate on the surface, with size and density increasing over time. At 600°C, the oxide layer on the films consisted of an inner aluminum oxide layer and an outer chromium oxide layer, mainly driven by the inward oxygen diffusion. After oxidation at 700°C and 800°C, the oxide layer evolved into a three-layer structure with an inner aluminum oxide layer, a middle chromium oxide layer, and an outer aluminum oxide layer. With prolonged oxidation time, the outer aluminum oxide layer developed an island-like structure with a discontinuous thickness. After extended oxidation at 800°C, form chromium oxide within and on the surface of the aluminum oxide. Additionally, internal oxidation of aluminum occurred inside the film.

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

Hard and Nanostructured Coatings III

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

8:20am **MA3-3-WeM-2 Comparative Study of the Effect of W and Nb Addition on Microstructure and Properties of Zr-Cu-Based Thin-Film Metallic Glasses***,* ***Deepika Thakur [deep0808@kfy.zcu.cz]****, Michaela Červená, Radomír Čerstvý, Petr Zeman,* University of West Bohemia - NTIS, Czechia

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

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

A systematic investigation revealed that W and Nb additions have a significant impact on microstructure and other properties of the films. The films remain amorphous with smooth surfaces (roughness < 2 nm) up to 65 at.% of W or Nb, displaying vein-like features typical of metallic glasses upon fracture. W-Zr-Cu films with 67 at.% W are characterized by a combination of featureless structures (amorphous-like) close to the substrate and thin columns in the upper part of the film. Films with even higher W contents grow in a V-shaped columnar microstructure corresponding to the bcc α-W crystalline structure. Nb-Zr-Cu films with 70 at.% Nb clearly exhibit a dual-phase structure with thin columns surrounded by vein-like features. Further increase in the Nb content above 70 at.% leads to the formation of a crystalline structure with parallel columns and very small voids. These voids tend to vanish with increasing Nb content. A gradual increase in hardness and reduced Young's modulus is observed with increasing W content for the amorphous W-Zr-Cu films and the crystalline films show an enhancement in hardness of up to 15% compared to films with pure W due to solid solution hardening. In the case of Nb-Zr-Cu films with up to 70 at.% Nb, the hardness remains nearly constant. However, further addition of Nb results in a decreased hardness and this reduction might be attributed to a less dense structure of the films.

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

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

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

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

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

9:00am **MA3-3-WeM-4 Influence of Bilayer Periodic Thickness Ratios on the Mechanical Properties and Corrosion Resistance of Alcrnbsitin/Alcrn High-Entropy Alloy Nitride Multilayer Thin Films**, ***Shang-Hua Tseng [bnb515032@gmail.com]****,* National Taiwan University of Science and Technology, Taiwan*; Jyh-Wei Lee,* Ming Chi University of Technology, Taiwan*; Chaur-Jeng Wang,* National Taiwan University of Science and Technology, Taiwan*; Bih-Show Lou,* Chang Gung University, Taiwan

High entropy alloy (HEA) nitride thin films have attracted considerable attention from the global industrial and academic communities due to their excellent mechanical properties. HEA multilayer nitride films also exhibit good interfacial stability, outstanding mechanical performance, and superior corrosion resistance. In this study, AlCrNbSiTiN/AlCrN nitride multilayer thin films were deposited using a high power impulse magnetron sputtering (HiPIMS) system with AlCrNbSiTi and AlCr targets in a mixed argon and nitrogen atmosphere. By adjusting the residence time of the substrates in the plasma regions of the AlCrNbSiTi and AlCr targets, multilayered thin films with varied bilayer periodic thicknesses ranging from 6 to 40 nm were fabricated. For the multilayer thin film with 15 nm bilayer period thickness, the thickness ratios of AlCrNbSiTiN and AlCrN single layer were adjusted to evaluate their influence on the hardness and corrosion resistance of films. XRD analysis indicated that all AlCrNbSiTiN/AlCrN multilayer films, as well as single-layer AlCrNbSiTiN and AlCrN films, exhibited a face-centered cubic crystal structure. Notably, the AlCrNbSiTiN/AlCrN multilayer film with a 15 nm bilayer period demonstrated a high hardness of 28 GPa and excellent corrosion resistance in 0.5 M H_2SO_4 aqueous solution, with a corrosion impedance value of 1.19 × 10^6W.cm^2. The influence of AlCrNbSiTiN to AlCrN thickness ratios on the mechanical properties and corrosion resistance of AlCrNbSiTiN/AlCrN multilayer thin film with 15 nm bilayer period was explored in this work.

9:20am **MA3-3-WeM-5 Impact of Microstructural Characteristics of HVOF-Deposited Cr3C2-Cermet Coatings on Their Performance in Sliding Abrasive Wear**, ***Xinqing Ma [Chin.ma@cwst.com]****, Peter Ruggiero,* Curtiss-Wright Corporate, USA

Nowadays, Cr_3C_2-based cermet hardface coatings manufactured by advanced HVOF processes are well recognized for their corrosion and erosion resistance, particularly at high temperatures. Their lightweight nature and high temperature capability make them an attractive alternative to WC-based alloy coatings and hard Cr plating coatings. The objective of this study is to develop optimal Cr_3C_2-NiCr coatings by comparing different feedstock materials, including feedstock with nanocrystalline and/or submicron sized Cr_3C_2 phases. The focus of the investigation is on understanding the impact of feedstock features such as particle size, morphology, and nanocrystalline carbide sizes, as well as sliding abrasive wear conditions on the coating properties and sliding wear performance. The results of the study indicate that the sliding wear resistance of the Cr_3C_2-NiCr coatings is highly influenced by the features of the Cr_3C_2 carbides. With the special interest of nano-crystaline and or submicron-sized carbides, the presence of nano, submicron and a few microns sized carbides in the coatings was revealed to improve their density, residual stress and hardness, leading to a significant reduction in wear rates under test conditions. Furthermore, the size of the abrasive SiC grit on the counter surface plays a significant role in determining the sliding wear behavior of these coatings. Based on the analysis of the test data, the mechanisms behind the performance of the Cr_3C_2-NiCr coatings have been investigated and used to interpret their sliding wear behaviors. This study has identified and recommended optimized materials for improved coating properties based on the key findings and results analyses. These findings and model analyses contribute to the understanding of the relationship between feedstock features, sliding abrasive wear conditions, and the wear rates of HVOF-sprayed Cr_3C_2-NiCr coatings. Hence, the optimized manufacture method by advanced HVOF method will meet the on-going need for a robust alternative solution to hard chromium plating (HCP) method.

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

Owing to its excellent mechanical properties TiSiN is commonly used as hard protective coating in cutting applications. However, the detailed investigation of the microstructure of TiSiN is a challenging task due to its nanocomposite structure, typically consisting of nanocrystalline and amorphous regions. Atom probe tomography would be a valuable method to study the local elemental distribution with high resolution, but peak overlaps of Si and N in the mass spectrum do not allow for an unambiguous differentiation, resulting in poor elemental accuracy and imaging precision. In order to improve both, isotopic substitution of naturally abundant nitrogen with ^{15}N enriched nitrogen was applied, allowing to disentangle the contribution of Si and N to the mass spectrum. In addition, the bulk composition of TiSiN coatings deposited with naturally abundant nitrogen was corrected by peak decomposition considering the corresponding isotopic abundancies, resulting in an improved elemental accuracy. A spatially resolved approach via voxeling the 3D reconstructed data and subsequent peak decomposition of the individual voxels also allows the improvement of the imaging precision. The results showed, that Si is to some extent incorporated into a $Ti_{1-x}Si_xN$ solid solution, but also Ti is incorporated into the amorphous $Si_xTi_yN_z$ phase fraction.

11:20am **MA3-3-WeM-11 Sputter Deposition of Ultrathick Boron Carbide Coatings on Rolling Spherical Substrates**, ***William Rios Lopez [rioslopez1@llnl.gov]****, James Merlo, Greg Taylor, Jean-Baptiste Forien, Sergei Kucheyev,* Lawrence Livermore National Lab, USA

Advancing inertial confinement fusion (ICF) technology requires the development of novel vapor deposition processes for the fabrication of spherical ablators. Boron carbide (B4C) is a promising material for next-generation ICF fuel ablator capsules due to its unique properties, enabling optimal ICF implosion dynamics. Furthermore, its compatibility with direct-current magnetron sputtering (DCMS) offers scalability for ablator production. However, the deposition of B4C films on spherical substrates remains challenging due to the complex interplay between directional deposition processes and substrate geometry. This study focuses on minimizing the density of nodular defects in ultrathick B4C coatings deposited by DCMS on rolling spherical substrates. We used a custom-designed substrate holder to systematically study how the rolling kinetics of spherical substrates influences the nodular defect population in coatings. This work was performed under the auspices of the U.S. DOE by LLNL under Contract DE-AC52-07NA27344.

11:40am **MA3-3-WeM-12 Superhard Single-phase $Ti_{1-x}Al_xB_y$ Films with Good Oxidation Resistance Grown without External Heating using Hybrid HiPIMS/DCMS Technique**, ***Bartosz Wicher [bartosz.wicher@liu.se]***, Linköping University, IFM, Thin Film Physics Division, Poland; *Vladyslav Rogoz,* Linköping University, IFM, Thin Film Physics Division, Ukraine; *Oleksandr Pshyk,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland, Ukraine; *Szilard Kolozsvari, Peter Polcik,* Plansee Composite Materials GmbH, Germany; *Ivan Petrov,* University of Illinois at Urbana-Champaign, USA, Bulgaria; *Lars Hultman, Grzegorz Greczynski,* Linköping University, IFM, Thin Film Physics Division, Sweden

A hybrid High-Power Impulse Magnetron Sputtering (HiPIMS) and Direct Current Magnetron Sputtering (DCMS) approach with TiB_2 and AlB_2 targets is used to grow $Ti_{1-x}Al_xB_y$ thin films with $0.40 \le x \le 0.76$ and $1.81 \le y \le 2.03$. The hybrid sputtering method ensures precise control over the energy and momentum of ionized species. The primary aim is to optimize the Al content for enhancing the high-temperature oxidation resistance while maintaining excellent mechanical properties that stem from the diboride structure. No external substrate heating is used resulting in the substrate temperature lower than 180°C.

Oxidation tests performed at temperatures ranging from 700 to 900 °C indicate a substantial improvement in oxidation resistance with higher Al content. Films with $x \le 0.49$ develop porous, B-depleted oxide layers exhibiting titanium dioxide (TiO_2) rutile phase structure and often show spallation. In contrast, the $Ti_{1-x}Al_xB_y$ layers with $x \ge 0.58$ form dense oxide scales composed of amorphous alumina (Al_2O_3) and borate ($Al_{18}B_4O_{33}$) phases, which effectively passivate the film surface against further oxidation. The oxide scales formed on the high-Al content films exhibit nanoindentation hardness comparable to that of TiAlN coatings and very good adhesion to the underlying substrate due to better matching of thermal expansion coefficients.

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

Acknowledgment

Bartosz Wicher is grateful to the ÅForsk Foundation for personal travel grant (ref.nr 24-721).

12:00pm **MA3-3-WeM-13 Nanostructured Multilayer Cr/C Coatings for Advanced Protection of Mold Tools**, *Yavor Sofronov,* ***Krassimir Marchev [k.marchev@northeastern.edu]***, *Milko Angelov, Milko Yordanov, Rayna Dimitrova, Krum Petrov, Antonio Nikolov, Todor Gavrilov,* Technical University of Sofia, Bulgaria

Nanostructured Cr/C multilayer coatings have been deposited by non-reactive unbalanced magnetron sputtering on DIN 1.2363 (A2) cold-worked air-hardened steel samples using proprietary system from Cr and graphite targets. Smooth crack-free coatings with bi-layer periods of 4.8, 3.1 and 2.2 nm have been produced by precision control of substrate rotation speeds of 2, 3, and 4 rpm.The high sp3/sp2 hybridization ratio, moderate hardness and high elastic recovery are essential for variety of industrial applications. Used deposition technology demonstrates high reliability and reproducibility of coatings.

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

High Entropy and Other Multi-principal-element Materials II

Moderators: Jean-François Pierson, IJL - Université de Lorraine, France, **Frederic Sanchette**, Université de Technologie de Troyes, France

8:00am **MA4-2-WeM-1 Oxidation Resistance of High Entropy Nitride Thin Films Deposited by Magnetron Sputtering**, *Djallel Eddine Touaibia, Abdelhakim Bouissil, Sofiane Achache, Mohamed El Garah,* ***Frederic Sanchette [frederic.sanchette@utt.fr]***, Université de Technologie de Troyes, France **INVITED**

In the last decades, Refractory High Entropy Alloys (RHEAs) thin films have attracted more attention owing to their enhanced mechanical properties and better thermal stability at high temperatures, compared to conventional alloys. TiTaZrHfW-N and TiTaZrHfAl-N RHEAs thin films were deposited by reactive magnetron sputtering technology at different N2 flow rates. For both systems, nitrogen-free films are amorphous, and the nitrides are columnar, single-phased with an FCC-NaCl type structure. The strong Me-N bonds lead to hardness up to 29 GPa and a Young's modulus up to 257 GPa, for the TiTaZrHfW-N system whereas the highest hardness and Young's modulus for the TiTaZrHfAl-N system are 25.3 GPa and 201.3 GPa respectively. Unlike metallic films, TiTaZrHfW-N and TiTaZrHfAl-N nitride films are thermally stable at 800 °C under vacuum and have a much better oxidation resistance. Nanolayered architectures TiTaZrHfW-N/Si3N4 and TiTaZrHfAl-N/Si3N4 result in a significant improvement of the oxidation resistance at 800 °C due to the formation of amorphous Si-N barrier nanolayers, hindering the oxygen diffusion.

8:40am **MA4-2-WeM-3 Plasmonic Behaviour of Multi-Component Nitride $(TiVZrNbTa)N_X$ Thin Films**, ***Miguel Piñeiro [miguel.pineiro-sales@univ-lorraine.fr]***, Institut Jean Lamour - Université de Lorraine, France, Peru; *Salah-Eddine Benrazzouq,* Institut Jean Lamour - Université de Lorraine, France, Morocco; *Valentin Milichko, David Pilloud, Thomas Easwarakhanthan,* Institut Jean Lamour - Université de Lorraine, France; *Frank Mücklich,* Saarland University, Germany; *Jean-François Pierson,* Institut Jean Lamour - Université de Lorraine, France

Although transition metal nitrides such as TiN or ZrN have been widely studied as plasmonic properties, the optical and electrical properties of multi-component nitride thin films are rather lacking in the literature for plasmonic applications, in spite of their well-known mechanical properties [1]. We attempt in this paper to alleviate this drawback by depositing the multi-component nitride $(TiVZrNbTa)N_x$ films on silicon substrates by using reactive magnetron sputtering under different nitrogen flow rates (R_{N2}) and at different working pressures. The X-ray diffractograms of the as-deposited films have shown that they crystallize in a single-phase with a rocksalt-like structure. Moreover, the plasmonic potential of the films was investigated from their dielectric function determined by variable angle spectroscopic ellipsometry. The films prepared at low working pressures exhibit optimal metallic behaviour with the real part of their dielectric function displaying zero-crossover. In contrast, those fabricated at high working pressure show non-metallic behaviour without any zero-crossover and with low absorption in the near-infrared region. In particular, the real part of the dielectric function of the film produced at 0.5 Pa has a notable feature of double epsilon-near-zero (2ENZ) comparable to other transition metal nitrides such as TiN, ZrN and NbN [2-4]. The dielectric function of $(TiVZrNbTa)N_x$ can be tuned by tailoring the deposition parameters such as working pressure to some desired plasmonic application. Specifically, the screened plasma energy (E_{ps}) is tuneable from near UV to visible ranges, from 3.4 to 2.1 eV. Their plasmonic performance were evaluated by calculating their intrinsic quality factor for surface plasmon polaritons (SPP). Additionally, the free-carrier density, the scattering time and the electrical resistivity of the films were also determined by means of Drude model for the free charge carrier contribution to the dielectric function [2]. Drude parameters were compared with additional electrical measurements performed by four-point probe method.

References

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9:00am **MA4-2-WeM-4 Temperature Stability of High Entropy Ceramic Cr-Hf-Mo-Ta-W-N Refractory Metal Coatings***, **Pavel Soucek [soucek@physics.muni.cz]**, Stanislava Debnarova, Matej Fekete,* Masaryk University, Czechia*; Sarka Zuzjakova,* University of West Bohemia, NTIS, Czechia*; Shuyao Lin,* Technische Universitat Vienna, Austria*; Ondrej Jasek, Tatiana Pitonakova,* Masaryk University, Czechia*; Nikola Koutna,* Technische Universitat Vienna, Austria*; Petr Zeman,* University of West Bohemia, NTIS, Czechia

High entropy alloys (HEAs) are multi-component materials composed of five or more principal elements, with each element's content ranging from 5 to 35 atomic percent. The properties of HEAs arise from four core effects: the high entropy effect, severe lattice distortion, sluggish diffusion, and the cocktail effect. This high entropy concept also extends to ceramics, including oxides, nitrides, borides, and carbides.

In this study, we are examining the temperature stability of high entropy nitrides from the Cr-Hf-Mo-Ta-W-N system. We utilized magnetron co-sputtering of segmented elemental targets for all depositions, which were performed on silicon and sapphire substrates. The first set of depositions was conducted at ambient temperature, while an elevated temperature of 700°C was used for the second set to enhance coating crystallization.

All the deposited coatings exhibited strong diffraction peaks corresponding to a face-centred cubic (fcc) lattice, which is anticipated for the formation of these high entropy ceramics. The coatings were annealed at temperatures of 1000°C, 1200°C, and 1400°C to observe changes in their chemical composition, phase, crystal structure, morphology, and mechanical properties.

We will discuss the significant role of coating adhesion in withstanding annealing, the impact of nitrogen loss on changes in the coating structure, and the influence of the inherent multilayered structure of the coatings on phase emergence and stability. Furthermore, we will identify critical elements that enhance the temperature stability of the coatings and discuss the limits of high entropy stabilization in the studied nitride system.

11:00am **MA4-2-WeM-10 The Microstructure, Mechanical Properties and Performance of High–Entropy (AlCrTiMoVNi)N Coatings Produced by Cathodic Arc Evaporation***, **Qi Yang [qi.yang@nrc-cnrc.gc.ca]**,* National Research Council of Canada*; Alex Lothrop, Xiao Huang,* Carleton University, Canada

High-entropy (AlCrTiMoVNi)N coatings were prepared using cathodic arc evaporation. The target composition was varied to investigate the effect of nickel concentration on the microstructure, mechanical properties and tribological performance of the coatings. All coatings assume a B1 face centered cubic structure, and contain many small droplet and large splat defects; and the amounts of those defects increase with the concentration of Ni. All coatings showed excellent high-temperature phase stability. The hardness and elastic modulus of the coatings reached maximum values at 2% Ni and then, decreased as the Ni content increases. In terms of performance, the coating with 2% Ni had the lowest wear rate while in erosion testing the coating free of Ni had the lowest erosion rates. Overall, the presence of droplets/splats had a significant influence on the tribological performance of the coating.

11:20am **MA4-2-WeM-11 Fabrication of CrMoNbWTi High Entropy Alloy Thin Films: Effect of Ti Content***, **Han-Jie Chen [youtude8@gmail.com]**,* Ming Chi University of Technology, Taiwan*; Bih-Show Lou,* Chang Gung University, Taoyuan, Taiwan*; Jyh-Wei Lee,* Ming Chi University of Technology, Taiwan

In 2004, Professor Yeh and Professor Cantor independently introduced innovative material systems known as high entropy alloys (HEAs) and multicomponent alloys. Due to their exceptional mechanical and physical properties, HEAs and their thin films have garnered significant attention from industrial, academic, and research communities. This study explores the fabrication and characterization of CrMoNbWTi HEA thin films grown on stainless steel (AISI 304 and AISI 420) and silicon substrates by high power impulse magnetron sputtering (HiPIMS) technology. The effect of titanium content on the phase, microstructure, mechanical properties, and corrosion resistance of CrMoNbWTi HEA thin films was studied. The films' cross-sectional morphology was analyzed using field emission scanning electron microscopy, while X-ray diffraction (XRD) was used to evaluate the crystalline structure. The mechanical properties, including hardness, elastic modulus, adhesion, and wear resistance, were evaluated through nanoindentation, scratch testing, and pin-on-disk wear testing. We can find out that the hardness of CrMoNbWTi HEA thin films increased from 20.7 GPa to 23.5 GPa with increasing Ti content. The adhesion critical load also increased with increasing Ti contents. The corrosion resistance of CrMoNbWTi HEA thin films also showed a positive tendency with Ti concentration. Due to its solid solution hardening and grain refinement effects, we can conclude that the Ti content plays an important role in the mechanical and corrosion resistance enhancement of CrMoNbWTi HEA films.

11:40am **MA4-2-WeM-12 Effect of Elemental Concentration on Mechanical and Tribological Properties of (AlNbSiTiZr)N Thin Films***, **Tongyue Liang [tongyue.liang@mail.mcgill.ca]**, Stéphanie Bessette, Raynald Gauvin, Richard Chromik,* McGill University, Canada

(AlNbSiTiZr)N thin films were deposited on silicon wafer and steel substrates using pulsed DC magnetron sputtering with four distinct targets (AlSi, Ti, Nb, Zr). The elemental concentration of each constituent was tuned by adjusting the discharge current applied to each target. The thickness of the deposited films was maintained at approximately 1.3 µm. Three different (AlNbSiTiZr)N thin films with slight variations in elemental concentrations were studied to assess the impact of compositional changes on their structure and properties. The films were characterized for surface and cross-sectional morphology, microstructure, roughness, and mechanical properties. A minor increase in the concentrations of Nb and Zr (5 at.% for each) led to a significant improvement in hardness, increasing from 12.7 ± 0.7 GPa to 20.8 ± 0.5 GPa. The tribological properties of the films were studied using a ball-on-plate tribometer under dry air conditions with a load of 0.5 N for 1000 sliding cycles. The results indicate that the wear resistance of the (AlNbSiTiZr)N thin films improved with the increased concentrations of Nb and Zr.

Protective and High-temperature Coatings
Room Palm 5-6 - Session MA5-1-WeA

Boron-containing Coatings I
Moderator: Anna Hirle, TU Wien, Austria

11:00am **MA5-1-WeA-10 Metal Boride Nanocrystal Inks for Applications in Extreme Environments***, **Loredana Protesescu [l.protesescu@rug.nl]**,* RUG, Netherlands **INVITED**

How can boron-rich nanocrystalline films be optimized to meet the stringent mechanical demands of extreme environment applications?

Modern advances in clean energy, hypersonic travel, and nuclear technologies place extraordinary demands on materials' thermal and mechanical durability. High-stakes fields, such as aerospace and space exploration, require materials that withstand extreme conditions, often exceeding 4,000 °C, with substantial mechanical strength and oxidation resistance. Refractory materials like ultra-high temperature ceramics (UHTCs), while promising, are limited by high production costs and challenging synthesis processes. This study seeks to address this challenge by exploring nanoscale metal boride materials—specifically, strontium hexaboride (SrB6) nanocrystals (NCs)—as a cost-effective, mechanically robust alternative.

Nanocrystals (NCs) offer unique advantages due to their high surface area, tunable crystallization, and the ability to form films with nanoscale precision, which is critical for enhancing mechanical properties in thin coatings. Here, we investigate the potential of surface-modified SrB6 NCs, blade-coated onto silicon and sapphire substrates, as a pioneering solution for boron-rich, super-hard thin films. Through ligand modification with BF4 and BI3, these NCs achieve distinct structural formations on different substrates, significantly impacting their mechanical performance.

Our findings demonstrate that SrB6-BI3 films on silicon reach up to 10 GPa hardness and a Young's modulus between 180 and 200 GPa. In comparison, SrB6-BF4 films attain 5 GPa hardness and 170 GPa modulus on silicon, with a notably higher modulus of 300 GPa on sapphire, suggesting enhanced stiffness through substrate optimization. Atomic force microscopy (AFM) revealed crystallization patterns where SrB6-BI3 formed micron-sized crystals on silicon, while SrB6-BF4 created spherical clusters, further affecting mechanical properties.

This study highlights that by optimizing ligand choice, substrate selection, and minimizing defects, boron-rich metal boride nanomaterials can be tailored for demanding applications. These findings position SrB6 NC-based films as a promising, cost-efficient alternative to conventional super-hard materials like diamond, with potential breakthroughs in extreme environment applications.

11:40am **MA5-1-WeA-12 Dos and Don'Ts When Performing Theoretical Predictions for Identification of Stable Metal-Boride Materials (MAN Phases)**, ***Martin Dahlqvist [martin.dahlqvist@liu.se]***, Linköping University, IFM, Materials Design Division, Sweden

The number of atomically laminated boron-based materials (MAB phases) have grown significantly since their discovery in the 1960s and in the last decade we have seen the realization of their two-dimensional derivatives, boridene. MAB phases are versatile in terms of chemical composition, which facilitate controlled and tailored properties. Widening and enhancement of these materials requires an enlarged palette of compositions. Alloying through the addition of elements is one way for expanding the compositional space of MAB phases and, in turn, their attainable properties. This has traditionally been realized through solid solutions upon metal alloying and recently through the formation of chemically ordered metal layers. This is where theory can be used for accelerating the exploration of next-generation MAB phases. It will be demonstrated how predicted stability can be used to identify the most promising novel material candidates to be used as guidance for synthesis experiments. The importance of considering both chemically ordered structures and disordered solid solutions for reliable predictions will also be discussed. is required. Results will cover ternary and quaternary MAB phases, the latter with metal disorder and order (in-plane *i*-MAB and out-of-plane *o*-MAB).

Protective and High-temperature Coatings
Room Town & Country D - Session MA4-3-WeA

High Entropy and Other Multi-principal-element Materials III

Moderators: Jean-François Pierson, IJL - Université de Lorraine, France, **Pavel Soucek**, Masaryk University, Czechia

2:00pm **MA4-3-WeA-1 Few-Layered Multi-Transition Metal Chalcogenide Heterostructured Alloy Absorber for High-Performance Photodetector***, **Chia-Ying Su [neowwow1114@gmail.com]**,* National Cheng Kung University, Taiwan*; I-Hsi Chen, Jyh-Ming Ting,* National Cheng Kung University (NCKU), Taiwan

Few-layered MoWSSe alloy with composition spread was synthesized using salt-assisted atmospheric pressure chemical vapor deposition. A heterojunctionphotodetector device was then made by connecting two electrodes to two areas that across a composition gradient. Basic material characteristics the photodetectorperformance were examined. We demonstrate that the photodetector exhibits the highest performance under visible light among a wide range of the incident light, witha responsivity greater than 10 A/W, detectivity over 5×10 10 jones, and external quantum efficiency exceeding 3000%. Even in the near-infrared wavelength range,the device still shows a responsivity greater than 1 A/W, detectivity over 5×10 9 jones, and external quantum efficiency over 150%. The rise time was also less than 5milliseconds. The outstanding performance of this photodetector device is attributed to the multiple p-n heterojunctions formed within a few-layered composition-gradientMoWSSe alloy, generating an internal electric field that facilitates the separation of photo-generated electron-hole pairs.

2:20pm **MA4-3-WeA-2 Sputter Deposition of Ta-W-Au-Bi High Entropy Alloys for Inertial Confinement Fusion Hohlraums***, **Daniel Goodelman [goodelman1@llnl.gov]**,* Lawrence Livermore National Laboratory, USA*; Nikhil Vishnoi, Gregory Taylor, Eunjeong Kim, Alison Engwall-Holmes, Swanee Shin, David Strozzi, Brandon Bocklund, Scott Peters, Sergei Kucheyev, Leonardus Bimo Bayu Aji,* Lawrence Livermore Laboratory, USA

The hohlraum is a centimeter-scale sphero-cylindrical cannister used as the housing for a hydrogen fuel capsule in an indirect-drive inertial confinement fusion (ICF) target. The hohlraum is a critical component in increasing the ICF energy yield. Our simulations with the radiation hydrodynamics code LASNEX suggest that the fusion yield can be improved by using hohlraums made of Ta-W-Au-Bi high entropy alloys (HEAs). However, the magnetron sputter deposition of these HEAs with low porosity and submicron grains remains a challenge. Here, we examine how tailoring the main deposition process parameters, including the average plasma discharge power, working pressure, substrate bias, target-to-substrate distance, and substrate temperature, can be leveraged to enable the fabrication of Ta-W-Au-Bi films with a dense microstructure and high electrical resistivity, thus providing a promising path forward for the development of next-generation ICF targets.

This work was performed under the auspices of the U.S. DOE by LLNL under Contract DE-AC52-07NA27344 and was supported by the LLNL-LDRD Program under Project No. 23-ERD-005.

2:40pm **MA4-3-WeA-3 ADREnALINe : Accelerated Design of Revolutionary Entropy-Augmented, Lasting and Innovative NitridEs – First Results on Oxidation Resistance of Binary and Ternary Nitrides***, Ludovic Méreaux,* IRCER, France*; Edern Menou, Thomas Vaubois,* SAFRAN, France*; Cédric Jaoul,* IRCER, France*; **Marjorie Cavarroc [marjorie.cavarroc@safrangroup.com]**,* SAFRAN, France

Increasing aircraft engine temperature is one method, amongst others, to decarbonize aviation. But at high temperature, *e.g.*, 1200 °C, 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 a data-driven screening tools 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.

Due to considered temperatures, coatings based on refractory elements such as Zirconium (Zr), Niobium (Nb), Molybdenum (Mo), Hafnium (Hf) ; Tantalum (Ta), Tungsten (W), Ruthenium (Ru) or Rhenium (Re), combined with Carbon (C), Nitrogen (N) or Boron (B), are credible potential candidates [1]. Cheaper and more abundant elements, as Iron (Fe) and Aluminium (Al), could also be considered in the mix to comply with industrial and environmental constraints.

High Entropy Alloys (HEA) (or Complex Concentrated Alloys (CCA) for their multiphase counterparts) are single-phase multielementary alloys showing original combination of properties (chemical resistance, mechanical resistance...) over a wide temperature range. The relatively new paradigm of HEA design, translated into the space of ceramics, offers new opportunities to meet high temperature requirements [2].

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

In this talk, we will highlight our methodology to combine numerical and experimental studies. First results about binary and ternary nitrides will be shown, together with the prospective work to come.

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3:00pm **MA4-3-WeA-4 Effect of Substrate Bias on Structural and Mechanical Properties of (MoNbTaW)N Coatings Deposited by Reactive DC Magnetron Sputtering***, **Saikumar Katta [saikumar.uoh@gmail.com]**,* University of Hyderabad, India

MoNbTaW is well known for its refractory high entropy properties which can maintain the same crystal structure even at very high temperatures without losing its mechanical properties. Nitrides of such (MoNbTaW)N will be a prime focus to get a hard and tough, mechanically stable high temperature withstanding coatings at room temperature.

In this study, (MoNbTaW)N hard coatings were deposited using a DC magnetron sputtering technique at a working pressure of 0.3Pa by varying substrate bias voltage from 0V to -200V. Optimized deposition parameters, including nitrogen flow and substrate temperature (400°C), were employed to produce dense and homogenous coatings on Silicon (100) substrates. X-Ray diffraction studies revealed that all the deposited films have Face Centered Cubic (FCC) crystal structure. A significant decrease in intensity ratio of principal reflection peak (111) to (200), from 2.39 to 0.84, is observed with increasing bias voltage from 0V to -200V. AFM studies indicated all the films have a fine granular morphology, with a maximum film thickness of 636nm at 0V, reducing to 550nm as the bias voltage is increased.

Topological analysis demonstrated that higher bias voltage led to smoother coatings, achieving an RMS roughness of < 2nm. XPS studies revealed that the covalency due to the increased bonding of p(N)-d(TM) with the increase in bias voltage. Nanoindentation studies confirmed a maximum hardness of 32 ± 2 GPa and a modulus of 345 ± 18 GPa at -200V bias. Additionally, the coatings displayed improved toughness, with the highest H/E value of 0.09 achieved at -200V.

3:20pm **MA4-3-WeA-5 Effect of Substrate Bias Voltage on Microstructure and Mechanical Behaviour of Equimolar VCrCoNi Alloy Thin-films Deposited via Unbalanced Magnetron Sputtering***, **Razie Hanafi [r.hanafi@unsw.edu.au]**,* UNSW, Australia*; Yujie Chen,* University of Adelaide, Australia*; Zhifeng Zhou,* City University of Hong Kong*; Zonghan Xie,* University of Adelaide, Australia*; Paul Munroe,* UNSW, Australia

Equimolar medium-entropy alloy VCrCoNi thin films were deposited on tool steel substrates by way of unbalanced magnetron sputtering, under different substrate bias voltages ranging from -20V to -120V. The deposited films were typically ~5.4 um thick. Variations in chemical composition as a function of bias voltage were observed, showing fluctuations in the concentrations of V, Ni, and Cr, while Co remained constant. These compositional variations arose from the interaction between the sputtered metal cations and the kinetic energy differences of the adatoms induced by changes in bias voltage. The thin films exhibited strong crystallographic textures and a microstructure characterized by ultrafine (< 5 nm) equiaxed grains. Changes in phase composition were also observed with variations in bias voltage. Hardness values ranged from 11 GPa to 14 GPa, peaking at -100 V bias. Additionally, scratch resistance and wear performance were examined, revealing correlations between microstructural characteristics and tribological behaviour.

3:40pm **MA4-3-WeA-6 Microstructure, Mechanical and Corrosion Properties of Reactively Sputtered (TiVCrZrNbMo)N High-Entropy Nitride Coatings***, **Žan Gostenčnik [zan.gostencnik@ijs.si]**, Aljaž Drnovšek, Matjaž Panjan, Matej Drobnič, Miha Čekada,* Jožef Stefan Institute, Slovenia

Since their discovery in 2004, high-entropy materials have been widely studied for their exceptional properties and broad application potential. Among these, high-entropy nitride coatings have emerged as promising candidates for protective coatings due to their superior mechanical and thermal properties. In particular, coatings containing refractory elements exhibit strong bonding with nitrogen, further enhancing their performance.

In this study, high-entropy nitride coatings composed of six refractory elements were synthesized with reactive direct current magnetron sputtering. The nitrogen flow ratio $R_N = N_2/(Ar + N_2)$ was varied from 0 to 50 % under a constant total gas flow to investigate the impact of nitrogen concentration on microstructure, crystal structure, mechanical properties, and corrosion resistance.

Microstructural and crystallographic analyses were conducted using X-ray diffraction (XRD), atomic force microscopy (AFM), and scanning electron microscopy (SEM). Elemental composition and chemical bonding were examined by X-ray photoelectron spectroscopy (XPS) and energy-dispersive spectroscopy (EDS). Mechanical properties were assessed using nanoindentation and profilometry, while corrosion resistance was evaluated using potentiodynamic polarization measurements.

XRD analysis revealed an amorphous structure for the coating without nitrogen, while nitride coatings exhibited a face-centered cubic (fcc) crystal structure. SEM imaging showed a columnar cross-section morphology. Hardness exceeded 30 GPa, while the reduced elastic modulus surpassed 250 GPa. Additionally, the coatings demonstrated enhanced corrosion resistance, highlighting their potential for protective applications.

4:00pm **MA4-3-WeA-7 High-Entropy Spinel Oxide Nanoparticles Achieve Record Low Thermal Conductivity and Diffusivity at High Temperatures***, **Yu Pei [y2pei@ucsd.edu]**,* University of California at San Diego, USA*; Renkun Chen, Ka Man Chuang, Sarath Adapa,* University of California San Diego, USA

Achieving efficient thermal insulation at high temperatures is critical for applications such as concentrating solar thermal (CST) and other thermal energy systems. Recent advancements in high-entropy ceramics offer a promising approach to tailoring thermal conductivity while maintaining excellent thermal stability. In this study, we demonstrate the realization of ultra-low thermal conductivity and diffusivity in ambient air using densely packed nanoparticle (NP) assemblies composed of high-entropy spinel oxides (HESOs) with more than five cation species. Unlike conventional porous thermal insulators, HESO-8 NP pellets achieve a high packing density while effectively suppressing all three major heat transfer mechanisms—solid conduction, gas conduction, and thermal radiation.

Our measurements reveal that the thermal conductivity of HESO-8 NP pellets remains as low as ~0.1 $W\ m^{-1}\ K^{-1}$ at high temperatures, approaching the conductivity of air. This remarkable reduction in heat transport arises from three key factors: (1) suppressed solid conduction due to minimal interparticle contact, (2) reduced gas conduction via nanoscale interstitial spaces, and (3) significantly attenuated thermal radiation enabled by the infrared-absorbing metallic spinel structure. Additionally, the relatively high packing density of these pellets results in much lower thermal diffusivity than aerogels, effectively delaying heat propagation under transient heat flux conditions.

Beyond their superior thermal insulation properties, the HESO NP pellets exhibit excellent thermal stability in air at elevated temperatures. Their high-entropy spinel structure resists coarsening (sintering), ensuring long-term stability in particle size and thermal performance. These findings highlight the potential of high-entropy oxide nanostructures as next-generation thermal insulation materials for high-temperature applications.

Protective and High-temperature Coatings
Room Palm 5-6 - Session MA5-2-ThM

Boron-containing Coatings II

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

8:40am **MA5-2-ThM-3 Influence of Boriding Treatment on the Tribological Performance of Tool Teel Repaired by Wire and Arc Additive Manufacturing**, ***Cesar Resendiz [resendiz.cesar@tec.mx]***, Tecnologico de Monterrey, Mexico

Interest in reconditioning metallic components has grown as a means to reduce industrial waste. Electric arc-based repair methods, including additive manufacturing techniques like Wire and Arc Additive Manufacturing (WAAM), impact microstructure and mechanical properties due to high heat input. Moreover, data on the reliability of components repaired through these methods under demanding tribological conditions remain limited. This study presents a tribological characterization of borided WAAM-repaired tool steel. To simulate tool damage, a groove measuring 6.35 mm in width and 3 mm in depth was created in AISI D2 steel samples using a spherical milling cutter. Material deposition was then manually performed by a certified technician using Gas Tungsten Arc Welding (GTAW) with ER308L wire, chosen for its availability and cost-effectiveness. Samples were subjected to three different conditioning treatments: (a) quenching and tempering followed by welding restoration (QTR treatment), (b) welding restoration followed by quenching and tempering (RQT treatment), and (c) welding restoration followed by boriding treatment (RB treatment). The physico-chemical characteristics of all samples were analyzed through optical microscopy and Raman spectroscopy. Mechanical surface properties were evaluated using instrumented indentation tests, while the adhesion of the boride coating on BT samples was assessed with VDI testing. Wear resistance and coefficient of friction (CoF) in both repaired and unrepaired regions of all sample types were measured using a micro-abrasion machine rig equipped with a load sensor. Wear scars were analyzed through scanning electron microscopy to identify dominant wear mechanisms. The results revealed that wear resistance in the repaired regions of QTR and RQT samples was significantly lower than that in their corresponding unrepaired regions. However, RB-treated samples exhibited consistent mechanical properties and tribological behavior, with improved wear resistance in both the repaired and unrepaired regions compared to QTR and RQT samples.

9:00am **MA5-2-ThM-4 Micromechanical Properties of $Ti_{1-x}Mo_xB_{2\pm z}$ Coatings Deposited by DCMS and HiPIMS**, ***Anna Hirle [anna.hirle@tuwien.ac.at]***, Christian Doppler Laboratory for Surface Engineering of High-performance Components,TU Wien, Austria*; Philipp Dörflinger, Rainer Hahn,* Christian Doppler Laboratory for Surface Engineering of High-performance Components, TU Wien, Austria*; Christian Gutschka, Tomasz Wojcik,* Christian Doppler Laboratory for Surface Engineering of High-performance Components, TU Wien,, Austria*; Maximilian Podsednik,* Institute of Chemical Technologies and Analytics, TU Wien, Austria*; Szilard Kolozsvári, Peter Polcik,* Plansee Composite Materials GmbH, Germany*; Carmen Jerg,* Oerlikon Surface Solutions AG, Liechtenstein*; Helmut Riedl,* Christian Doppler Laboratory for Surface Engineering of High-performance Components, Austria

A promising strategy for enhancing the limited fracture characteristics of sputtered transition metal diboride (TMB_2) thin films, including hardness and fracture toughness, is the formation of ternary diborides. Theoretical predictions based on density functional theory (DFT) indicate that Mo alloying in TiB_{2+z} may prove beneficial in reducing the inherent brittleness of such diboride coatings. The present study aims to provide experimental investigations of ternary $Ti_{1-x}Mo_xB_{2\pm z}$ coatings prepared by direct current magnetron sputtering (DCMS) and high-power impulse magnetron sputtering (HiPIMS) to validate the predictions and to investigate the influence of different deposition techniques.

A series of coatings was deposited using target compositions of TiB_2/C 99/1 wt. %, TiB_2/MoB 95/5 mol %, and TiB_2/MoB 90/10 mol %, resulting in coating compositions ranging from 0 at. % Mo to 4.7 at. % Mo. A variety of analytical techniques, including transmission electron microscopy (TEM), scanning electron microscopy (SEM), and X-ray diffraction analysis (XRD), were employed to characterize the microstructural properties. The chemical composition was determined by inductively coupled plasma optical emission spectroscopy (ICP-OES). To investigate the micromechanical properties of the ternary $Ti_{1-x}Mo_xB_{2\pm z}$ coatings, including hardness, fracture toughness, and fracture strength, nanoindentation, in-situ cantilever bending tests, and micropillar compression tests were employed.

The present study demonstrates that HiPIMS processes result in a considerable enhancement of hardness, fracture toughness, and fracture strength compared to DCMS. Specifically, the hardness of the HiPIMS coatings was enhanced from 38.8 ± 1.7 GPa to 43.7 ± 1.2 GPa, while the fracture toughness increased by 0.4 MPa√m and the $R_{p0.2}$ value rose by approximately 2 GPa. In comparison, the DCMS coatings exhibited a consistent decline in mechanical properties with increasing Mo content. Our findings highlight the significance of the energetics of growth conditions for novel ternary diboride systems.

9:20am **MA5-2-ThM-5 Tuning Properties of Diborides by Transition Metal Alloying Deposited by Combination of Magnetron Sputtering and Cathodic ARC Evaporation**, *Daniel Karpinski,* ***Keith Thomas [k.thomas@platit.com]****, Pavla Karvankova,* PLATIT AG, Switzerland*; Hannes Joost, Heiko Frank,* GFE-Schmalkalden e.V., Germany*; Pavel Soucek, Petr Vasina,* Institute of Physics and Plasma Technology, Masaryk University, Czechia*; Fedor Klimashin, Johann Michler,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland*; Jan Kluson,* PLATIT a.s., Czechia*; Christian Krieg, Andreas Lümkemann, Hamid Bolvardi,* PLATIT AG, Switzerland

Titanium diboride is currently themost widespreadmetal boride (MeB_x) coating used in industry due to itsoutstanding properties such as high hardness >40 GPa, high melting point >3000 °C, and low propensity for sticking to soft metals.The main drawbacks of diborides are their generally low oxidation resistance (<800°C for TiB_2) and brittleness. This study investigates the effect of alloying MeB_xwithtransition metalson the structure, mechanical and tribological properties, and oxidation resistance of the coating. The coating deposition was performed in a Platit Pi411 machinewith LACS® technology which includes simultaneous magnetron sputtering from a central cylindrical cathode (SCiL®) and a cathodic arc evaporation from cylindrical cathode located in the chamber door (LARC®). Here, the MeB_x target was sputtered, and cathodic arc evaporation of Ti or Cr target was used for alloying thecoating. XRD, HRTEM structure study, nanoindentation and isothermal annealing in air at Ta=600–900°C revealed that by alloying of MeB_xwe can form the nanolaminate microstructure, tune the hardness and modulus, and enhance oxidation resistance of the coating, respectively.

10:20am **MA5-2-ThM-8 Effect of Duty Cycle on the Microstructure and Mechanical Properties of Titanium Diboride Thin Films Deposited by High-Power Pulsed Magnetron Sputtering**, *Jian-Fu Tang,* National Kaohsiung University of Science and Technology, Taiwan Jian-Fu Tang, Taiwan*;* ***Min-Yi Lin [M12188007@o365.mcut.edu.tw]***, Department of Materials Engineering, Ming Chi University of Technology, Taiwan, ROC*; Fu-Sen Yang,* Department of Mechanical Engineering, National Taiwan University of Science and Technology, Taiwan, ROC*; Chi-Lung Chang,* Department of Materials Engineering, Ming Chi University of Technology, Taiwan, ROC

With the rapid advancement of modern technology, the growing development of 5G and artificial intelligence (AI) has led to a substantial increase in demand for printed circuit boards (PCB). Enhancing the performance of cutting tools used in PCB drilling has become essential to meet supply and application needs, especially in the application of non-ferrous metal materials. Surface treatments aimed at improving tool wear resistance, high-temperature durability, anti-adhesion properties, hardness, and overall lifespan are common strategies in the industry. Titanium diboride (TiB_2) and titanium diboride-based nitride films, known for their high hardness, excellent wear resistance, high-temperature stability, and thermal conductivity, are ideal coating materials for cutting tools.

This study used high-power impulse magnetron sputtering (HiPIMS) to investigate the microstructure and mechanical properties of TiB_2 films deposited under various duty cycles. Five samples were prepared under identical target power output (3.5 kW) and frequency (200 Hz), using different duty cycle settings: 3%, 5%, 10%, 25%, and DC.The analysis results indicate that the peak power density increases with the duty cycle decrease. Energy dispersive spectroscopy (EDS) analysis also confirmed that the film composition was consistent with the alloy target proportions. Nanoindenter analysis shows that as the duty cycle decreases, the hardness increases significantly, from 28.3GPa to 43.2GPa, and the residual stress increases, from -0.21GPa to -6.18 GPa. This can be attributed to the higher peak power density effect. In addition, all samples showed good adhesion (HF1~HF2), excellent wear resistance (< 8.4×10^{-7} $mm^3N^{-1}m^{-1}$), and lower friction coefficient (0.39 to 0.55), indicating that TiB_2 films have potential in PCB drilling application.

Keywords: TiB_2, duty cycle, high power impulse magnetron sputtering, hardness, residual stress

10:40am **MA5-2-ThM-9 TiB_2/Hf Superlattices: Exploring Mechanical Strength, Fracture Toughness, and Stress-Strain Behavior***, **Naureen Ghafoor [naureen.ghafoor@liu.se]**, Firat Angay, Marcus Lorentzon,* Linköping University, IFM, Sweden*; Rainer Hahn,* TU Wien, Austria, Sweden*; Michael Meindlhumer,* University of Leoben, Austria*; Lars Hultman, Jens Birch,* Linköping University, IFM, Sweden

We present experimental investigations on iso-structural TiB_2/Hf superlattices, exploring the impact of layer thickness on hardness, toughness, and fracture resistance. Ab initio calculations, which guided material selection, reveal a basal-plane lattice and shear modulus mismatch of 0.16 Å (5.4%) and 200 GPa between TiB_2 and Hf, hindering dislocation glide at interfaces. Superlattices were deposited with periods ranging from 2 to 10 nm and were characterized by XTEM, XRD, and ERDA for structural and compositional details. High hardness (40 GPa) and structural integrity were achieved at lower Hf thicknesses and higher growth temperatures, with boron diffusion from TiB_2 into Hf forming single-crystal TiB_2 and understoichiometric HfB_2. No strain buildup or epitaxial breakdown was observed in films with up to 375 periods, attributed to self-diffusion. Pillar compression tests were performed to measure stress-strain curves and determine fracture values of the superlattices. A strong correlation between the metallic Hf layer and ceramic TiB_2 was observed, with fracture stress varying across samples—some exhibited values up to 22 GPa, while others showed localized plasticity. Fracture stress showed minimal dependence on the superlattice period, suggesting that morphology did not significantly influence mechanical behavior, as all coatings exhibited similar column diameters. Plasticity was attributed to localized slip, which was visible in SEM and analyzed in TEM. Boron and oxygen diffusion in Hf layers impedes flow, increasing strength but reducing elongation, consistent with the Hall-Petch effect. The high strength of Hf restricts dislocation motion, particularly in thin superlattices, while TiB_2's single-crystal structure eliminates easy fracture paths, enhancing toughness. Assuming similar trends to nitrides, TiB_2/Hf superlattices are expected to exhibit significantly higher fracture stresses. Based on the Young's moduli of TiB_2 (~400 GPa) and Hf (~80 GPa), a maximum outer fiber strain of 4% in bending would result in ~16 GPa in TiB_2 and ~3.2 GPa in Hf. The Hall-Petch effect further strengthens the material, with flow stress estimates between 4.7-6.7 GPa in tension, while bending primarily affects the outer layers. The difference in Young's moduli between TiB_2 and Hf contributes to high fracture toughness, highlighting TiB_2/Hf superlattices as promising materials for applications requiring high hardness, toughness, and fracture resistance.

11:00am **MA5-2-ThM-10 Production of Thin Films of Cubic Boron Nitride with Almost No Residual Stresses by Pulsed Laser Deposition and Laser Stress Relaxation***, **Falko Jahn [jahn@hs-mittweida.de]**,* Mittweida University of Applied Sciences, Germany*; Thomas Lampke,* University of Technology Chemnitz, Germany*; Steffen Weissmantel,* Mittweida University of Applied Sciences, Germany

For decades boron nitride has been researched as a coating material due to its outstanding mechanical, thermal and chemical properties. Especially the cubic phase (c-BN) as second hardest material known so far with high thermal and chemical resistance has driven the desire to make this material usable for industrial applications. Pulsed Laser Deposition has been one of the few deposition techniques with deposition rates of several tens nm per minute, high enough for industrial needs [1]. However, like other deposition techniques, PLD produced c-BN-coatings show very high compressive intrinsic stresses which limits the film thickness to a few hundred nm.

We present a method to produce thin films of cubic boron nitride which contain almost no residual stresses. These thin films were deposited using Ion Beam Assisted Pulsed Laser Deposition as sublayers of 100 nm film thickness on silicon substrates. Applying a modified laser stress relaxation technique [2], we were able to reduce the intrinsic compressive stresses in these sublayers from 10 GPa to less than 1 GPa.

Alternating deposition and stress relaxation of such sublayers successively enables film thicknesses relevant for industrial applications, such as wear resistant coatings. One possibility is to stack relaxed pure phase c-BN-sublayers in order to obtain applicable cubic boron nitride coatings. Another possibility is the combination of c-BN-sublayers with hard h-BN-sublayers to form multilayer system with increased mechanical properties. These h-BN-sublayers produced by PLD show indentation hardnesses in the range of 15 – 25 GPa, which allows the whole multilayer system to be superhard with indentation hardness above 40 GPa.

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11:20am **MA5-2-ThM-11 Influence of Deposition Parameters on the Microstructure, Mechanical and Anti-Corrosion Characteristics of (Hfvtizrw)B2 High Entropy Alloy Boride Thin Films***, **Jun-Xing Wang [wangxing1470@gmail.com]**,* Ming Chi University of Technology, Taiwan*; Bih-Show Lou,* Chang Gung University, Taoyuan, Taiwan*; Riedl-Tragenreif Helmut,* Technische Universität Wien, Austria*; Jyh-Wei Lee,* Ming Chi University of Technology, Taiwan

In recent years, the exceptional mechanical and physical properties of highentropy alloy (HEA) boride thin films have garnered significant attention, sparking interest among the global industrial community, academia, and researchers. This study selected $(HfVTiZrW)B_2$ HEA boride as the target material to sputter $(HfVTiZrW)B_2$ HEA boride films under different substrate temperatures by high power impulse magnetron sputtering.

The results indicated that as the deposition temperature increases from 200°C to 500°C, the HEA boride films consistently exhibited a hexagonal close-packed (HCP) structure. Composition analysis and bonding energy assessments revealed that all films were metallic diborides.The highest hardness of 38.0 GPa was obtained for the film deposited at 500°C. The wear coefficient of friction was from 0.33 to 0.57. An excellent wear rate of 2.98×10^{-6} mm/N·m was achieved.

Observations from high-resolution transmission electron microscopy revealed that the grain size of the films increased from 11.39 ± 0.84 nm to 27.25 ± 3.59nm as deposition temperature increased from 200 to 500°C. The corrosion resistance of the HEA boride films in the 0.5 M H_2SO_4 aqueous solution was 38.89 times greater than that of 304 stainless steel. Furthermore, good oxidation resistance of HEA films in dry air atmospherebelow 600°C was observed. In summary, this study demonstratedthe $(HfVTiZrW)B_2$ HEA boride films exhibitedpromising applications as protective coatings for cutting tools and forming dies.

11:40am **MA5-2-ThM-12 Comparative Analysis of Oxidation Behavior and Mechanical Properties of $Hf_{0.24}Al_{0.06}B_{0.70}$ vs. $Hf_{0.35}B_{0.65}$ Thin Films***, **Eva B. Mayer [mayer@mch.rwth-aachen.de]**, Janani D. Ramesh,* RWTH Aachen University, Germany*; Zsolt Czigány,* Centre for Energy Research, Hungary*; Marcus Hans,* RWTH Aachen University, Germany*; Daniel Primetzhofer,* Uppsala University, Sweden*; Lukas Löfler, Jochen M. Schneider,* RWTH Aachen University, Germany

To improve the oxidation behavior of HfB_2 films for applications at high temperatures the addition of Al is a promising approach, however, at the expense of the mechanical properties, as the addition of 20 at.% Al causes a reduction in E-modulus by ~27 %. Therefore, the oxidation behavior and mechanical properties of magnetron sputtered $Hf_{0.35}B_{0.65}$ and$Hf_{0.24}Al_{0.06}B_{0.70}$ thin films are compared. ERDA, XRD and STEM data are employed to identify changes in composition, phase and oxide thickness, respectively, before and after isothermal oxidation in ambient air at 700°C for 1, 4 and 8 h each.

The addition of 6 at % of Al leads to the formation of an amorphous passivating oxide scale, exhibiting only 9.7 % of the scale thickness obtained on $Hf_{0.35}B_{0.65}$ after 8 h of oxidation. Nanoindentation measurements indicate that, the hardness and elastic modulus of $Hf_{0.24}Al_{0.06}B_{0.70}$ (35.6 ± 1.5 GPa and 501.4 ± 13.4 GPa) do not significantly differ from $Hf_{0.35}B_{0.65}$ (34.8 ± 1.0 GPa and 494.4 ± 13.8 GPa).

Hence, the incorporation of 6 at. % Al into HfB_2 improves the oxidation behavior by an order of magnitude, while the reduction in stiffness remains within the error range of the measurement.

Protective and High-temperature Coatings
Room Golden State Ballroom - Session MA-ThP
Protective and High-temperature Coatings Poster Session

MA-ThP-1 High Temperature Fracture Characteristics of Si Containing Ternary and Quaternary Transition Metal Diborides, ***Anna Hirle [anna.hirle@tuwien.ac.at]***, *Ahmed Bahr, Rainer Hahn, Tomasz Wojcik,* Christian Doppler Laboratory for Surface Engineering of High-performance Components, TU Wien, Austria; *Szilard Kolozsvári, Peter Polcik,* Plansee Composite Materials GmbH, Germany; *Jürgen Ramm, Carmen Jerg,* Oerlikon Surface Solutions AG, Liechtenstein; *Helmut Riedl,* Christian Doppler Laboratory for Surface Engineering of High-performance Components, TU Wien, Austria

To enhance the restricted oxidation resistance of transition metal diboride (TMB) ceramics, alloying with Si and disilicide phases is an effective method, resulting in the formation of highly dense and protective SiO_2 scales. This phenomenon has been well documented in the context of bulk ceramics [1, 2], and recent studies have also corroborated its occurrence in thin-film TMBs, including CrB_2, HfB_2, and TiB_2 [3, 4]. The incorporation of Si, $TaSi_2$ or $MoSi_2$ into TiB_2 results in a significant reduction in oxidation kinetics, while exhibiting only minor effects on the mechanical properties. In the case of quaternary TiB_2-based coatings, hardness values of 36 GPa ($TaSi_2$) and 27 GPa ($MoSi_2$) have been achieved, in comparison to approximately 38 GPa for the binary system. All of the aforementioned coatings exhibited α-AlB_2 crystal structure, with a preferred (0001) orientation being a key factor in achieving the highest hardness. Nevertheless, the fracture characteristics of these Si-alloyed TMBs remain largely unexplored.

The objective of the present study is to elucidate the fracture characteristics, particularly K_{IC}, of these Si-containing TMBs at elevated temperatures up to 850 °C through the application of in-situ micromechanical testing techniques. Accordingly, a series of Ti-TM-Si-$B_{2\pm z}$ coatings was deposited via non-reactive DC magnetron sputtering using a variety of composite targets, including TiB_2, $TiB_2/TiSi_2$ (90/10 & 80/20 mol%), $TiB_2/TaSi_2$ (90/10 & 80/20 mol%), and $TiB_2/MoSi_2$ (85/15 & 80/20 mol%). To gain a deeper understanding, additional detailed structural investigations were conducted using X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and elastic recoil detection analysis (ERDA).

In comparison to the binary TiB_{2+z} and the quaternary Ti-Ta-Si-$B_{2\pm z}$, the Si and $MoSi_2$-containing coatings exhibited a distinct onset of plastic deformation at approximately 600 °C. This phenomenon can be attributed to the precipitation of silicon-containing phases, which underlines the significance of conducting material testing at temperatures relevant to their intended applications.

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MA-ThP-3 Spinodal Decomposition and Nano-precipitate Formation in Ag-modified High-Entropy Alloys, ***Salah-eddine Benrazzouq [salah-eddine.benrazzouq@univ-lorraine.fr]***, *Abdelkrim Redjaimia, Jaafar Ghanbaja, Sylvie Migot, Valentin A. Milichko, Jean-François Pierson,* Institut Jean Lamour - Université de Lorraine, France

Phase separation in multi-component alloys presents both challenges and opportunities for material design. While traditionally viewed as a limitation, controlled phase separation could enable unique microstructural features and enhanced properties. High-entropy alloys (HEAs) have garnered significant attention across various research fields owing to their exceptional properties. This study investigates the distinctive behavior of silver addition to the CrMnFeCoNi Cantor alloy, where silver's higher mixing enthalpy creates an interesting case of spinodal decomposition and nano-precipitate formation.

Using DC magnetron co-sputtering, we synthesized CrMnFeCoNiAg thin films with systematically varied silver content. X-ray diffraction (XRD) patterns reveal distinct non-mixing behavior with the emergence of pronounced peaks corresponding to both silver and Cantor alloy phases. Cross-section bright-field TEM micrograph and SAED patterns revealed a dense structure with Ag precipitates dispersed throughout the 900-nm-thick film. HRTEM micrographs showed a nanoprecipitate morphology with fine-scale linear precipitates, while STEM-HAADF imaging highlighted the internal structure, revealing characteristic modulated patterns with striations parallel to the basal plane, indicative of spinodal decomposition with cuboidal particles and tweed-like contrast patterns.

The controlled formation of these nano-precipitates and their unique distribution pattern suggests potential for mechanical property enhancement through precipitation strengthening mechanisms. Our findings demonstrate how controlled phase separation can be used to engineer microstructure in HEA thin films. This understanding provides new strategies for designing multi-functional materials through deliberate exploitation of immiscibility effects, advancing our knowledge of phase evolution in complex alloy systems and offering pathways for property optimization in advanced coating applications.

MA-ThP-4 Influence of Si Content on Cracking Behavior of CrAlSiN Coatings, *Kirsten Bobzin, Christian Kalscheuer,* ***Max Philip Möbius [moebius@iot.rwth-aachen.de]***, *Jessica Borowy,* Surface Engineering Institute - RWTH Aachen University, Germany

The increasing demands for workpiece quality and cost-effectiveness in machining processes necessitate a comprehensive consideration of all relevant factors, including cutting parameters, materials, tool coatings, and geometry. Physical Vapor Deposition (PVD) manufactured CrAlSiN nanocomposite coatings, composed of CrAlN grains in a SiN_x matrix, represent a promising solution for improved tool life of milling tools. The elastic-plastic properties of the coating and the deformation behavior of the material composite thereby can be deliberately influenced by varying the silicon content.

CrAlSiN coatings with silicon contents of x_{Si} = 10, 15, 20, and 25 at.-% in the metal portion were fabricated on cemented carbide WC-Co substrates. The indentation hardness H_{IT} and indentation modulus E_{IT} of the coatings were measured through nanoindentation (NI) with a force of F_{NI} = 10 mN, using a Berkovich indenter. Additionally, crack resistance was evaluated using quasi-static high load (HL) nanoindentation tests under forces ranging from F_{HL} = 750 to 1,750 mN, with increments of ΔF_{HL} = 250 mN. A conical diamond indenter was used for the high load nanoindentation tests. The resulting indents were subsequently analyzed using scanning electron microscopy (SEM). The findings reveal that the indentation hardness H_{IT} remains unchanged at H_{IT} = (25.48 ± 1.59) GPa, while the indentation modulus increases with higher silicon content, ranging from E_{IT} = (222.64 ± 10.45) GPa for x_{Si} = 10 at.-% up to E_{IT} = (239.89 ± 7.78) GPa for x_{Si} = 20 at.-%. After high load nanoindentation all coatings exhibit no cracks at F_{HL} = 750 mN. With F_{HL} ≥ 1,000 mN on the other hand cracks can be observed in all coatings. Nevertheless, with rising silicon content, the maximum indentation depth h_{max} decreases, while the residual indentation depth h_0 remains constant. Furthermore, the proportion of plastic work shows a slight reduction as silicon content x_{Si} increases. These results indicate that the resistance against plastic deformation of the CrAlSiN coating increases with higher silicon content.

Coatings with high silicon content demonstrate promising resistance against plastic deformation at room temperature, highlighting their potential for further investigation. This initial test qualifies these coatings for additional studies under high-temperature conditions, aiming to enhance their applicability in machining processes. The insights gained from this research could lead to the development of more durable and efficient cutting tools, ultimately improving productivity in industrial applications.

MA-ThP-5 Relationship between Optical and Electrical Properties and the Microstructure of High Entropy Nitride (TiVZrNbTa)N_X Thin Films, ***Miguel Piñeiro [miguel.pineiro-sales@univ-lorraine.fr]***, Institut Jean Lamour - Université de Lorraine, France, Peru; *Salah-Eddine Benrazzouq,* Institut Jean Lamour - Université de Lorraine, France, Morocco; *Alexandre Bouché, Valentin Milichko, David Pilloud, Thomas Easwarakhanthan,* Institut Jean Lamour - Université de Lorraine, France; *Frank Mücklich,* Saarland University, Germany; *Jean-François Pierson,* Institut Jean Lamour - Université de Lorraine, France

In this study, high entropy nitride TiVZrNbTa thin films were prepared by DC reactive magnetron sputtering on silicon substrates at room temperature. The impact of varying nitrogen flow rateson the structural, microstructural, optical and electrical properties were investigated. X-ray diffraction technique revealed that all the deposited films exhibited a polycrystalline structure with fcc phase. However, the pure metallic samples displayed an amorphous structure [1]. Optical properties analysis showed a decrement of the reflectance compared with free-nitrogen sample in the infrared region, as determined by UV-VIS spectroscopy [2]. Hall-effect measurements indicate that the electrical resistivity for all samples remained within the range between 100 and 300 μΩ cm. Interestingly, samples deposited with applied substrate bias power during the deposition process did not show a significant change in resistivity. This suggests that substrate biasing has minimal effect on the electrical transport properties of the latter films. On the other hand, applying adjustable substrate bias led to a blueshift in the epsilon-near-zero (ENZ) wavelength. Furthermore, X-ray photoelectron spectroscopy (XPS) shows the effect of the nitrogen flow rate on the residual stress [3] and plasmon frequency. The impact of varying nitrogen flow rates on the microstructural properties were further investigated and explained.

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MA-ThP-6 Microstructure Evolution and Oxidation Behavior of Diffusion Pt-γ/γ' and Pt-aluminide Coatings at 1200 °C, ***Radoslaw Swadzba [radoslaw.swadzba@git.lukasiewicz.gov.pl]**, Agnieszka Sasiela,* Lukasiewicz Research Network - Uppersilesian Institute of Technology, Poland*; Boguslaw Mendala, Lucjan Swadzba,* Silesian University of Technology, Poland*; Lukasz Pyclik, Michal Gut,* Avio Polska sp. z o. o., Poland

This study examines the microstructural evolution and oxide scale growth of Pt-γ/γ' and Pt-aluminide diffusion coatings applied to a second-generation single-crystal Ni-based superalloy at 1200 °C. The Pt-γ/γ' coatings were produced through platinum electroplating followed by a 2-hour diffusion heat treatment at 1079 °C. Subsequently, Vapor Phase Aluminizing (VPA) at 1079 °C for 6 hours generated Pt-modified aluminide coatings. Both coating types were subjected to Thermogravimetric Analysis (TGA) in air for 20 hours as well as cyclic oxidation test up to 300 1-hour cycles at 1200 °C.

The initial and oxidized coatings were characterized using Electron Backscatter Diffraction (EBSD) to analyze phase transformations, grain size evolution, and interdiffusion between the coatings and the substrate alloy. In the as-deposited state, the Pt-γ/γ' coating consisted of γ and γ' grains enriched in Pt, with a thickness of approximately 27 µm, while the Pt-aluminide coating exhibited an outer zone of $PtAl_2$ and β-NiAl phases. During the high temperature oxidation testing, the Pt-γ/γ' coating showed grain growth and Pt diffusion to a depth of approximately 70 µm after 20 hours. The Pt-aluminide coating underwent martensitic transformation in its outer layer, with Al-depleted β-NiAl in its middle region and an interdiffusion zone containing Cr-rich precipitates.

High-resolution Scanning Transmission Electron Microscopy (STEM) provided detailed characterization of the alumina oxide scales formed on both coatings, revealing information on oxide grain size and the segregation of reactive elements (RE) to grain boundaries.

MA-ThP-7 Unprecedented B Solubility in Cubic (Hf,Ta,Ti,V,Zr)B-C-N Coatings, *Andreas Kretschmer,* TU Wien, Austria*; Marcus Hans, Jochen Schneider,* RWTH Aachen University, Germany*; **Paul Mayrhofer [paul.mayrhofer@tuwien.ac.at]**,* TU Wien, Institute of Materials Science and Technology, Austria

We investigate the influence of compositional complexity in Ti-B-C-N-based coatings by depositing Ti-rich (Hf,Ta,Ti,V,Zr)B-C-N coatings with varying B/C ratios (from 0/21 to 32/0 at%). Despite the high B content of 32 at% in the C-free material, this $(Hf_{0.1}Ta_{0.1}Ti_{0.6}V_{0.1}Zr_{0.1})B_{0.6}N_{0.4}$ forms a single-phase fcc solid solution without a boride tissue phase. All coatings—$(Hf_{0.1}Ta_{0.1}Ti_{0.6}V_{0.1}Zr_{0.1})B_xC_{0.5-x}N_{0.5}$ with x = 0.2, 0.3, 0.4, $(Hf_{0.1}Ta_{0.1}Ti_{0.6}V_{0.1}Zr_{0.1})B_{0.6}N_{0.4}$, and $(Hf_{0.1}Ta_{0.1}Ti_{0.6}V_{0.1}Zr_{0.1})C_{0.4}N_{0.6}$—exhibit similar hardness values of 37–38 GPa, but increasing B content leads to a decreasing indentation modulus. This trend is supported by *ab initio* calculations of fcc-$(Hf_{0.1}Ta_{0.1}Ti_{0.6}V_{0.1}Zr_{0.1})B_xC_{0.5-x}N_{0.5}$ (for x = 0, 0.125, 0.25, 0.375, 0.5), which also confirm the stability of these solid solutions over a wide compositional range. Despite increasing chemical complexity, the addition of B and C has little effect on lattice distortion.

Among the investigated coatings, $(Hf_{0.1}Ta_{0.1}Ti_{0.6}V_{0.1}Zr_{0.1})B_{0.4}C_{0.1}N_{0.5}$ provides the best balance between high hardness (37.7±1.0 GPa) and fracture toughness (K_{IC} = 4.0±0.5 MPa·m$^{0.5}$). This compositionally complex, single-phase, fcc-structured Ti-rich (Hf,Ta,Ti,V,Zr)B-C-N retains its hardness—which even slightly increases to 38.3±1.3 GPa—upon vacuum annealing up to 1200 °C. X-ray diffraction and atom probe tomography confirm its high-temperature phase stability, as an hcp-TiB_2-based phase forms only upon annealing beyond 1200 °C. More generally, all $(Hf_{0.1}Ta_{0.1}Ti_{0.6}V_{0.1}Zr_{0.1})B_xC_{0.5-x}N_{0.5}$ coatings with x = 0.2, 0.3, and 0.4 exhibit a total configurational entropy of ~1.1·R (~1.25·R at the metal sublattice and 0.95·R at the non-metal sublattice) and maintain a hardness of 36–38 GPa even when annealed at 1200 °C, contrary to compositionally simpler coatings, which soften to below 29 GPa.

These findings highlight the advantages of compositionally complex mixed ceramic coatings, which outperform simpler Ti-B-N or Ti-C-N coatings with similar structure and composition. Furthermore, they demonstrate how solubility limits can be extended beyond currently known boundaries through advanced materials science, enabling outstanding properties.

MA-ThP-8 Ab Initio Assessed Influence of Si on the Structural Integrity of Group IV Transition Metal Diborides, *Christian Gutschka, Lukas Zauner, Thomas Glechner,* Christian Doppler Laboratory for Surface Engineering of high-performance Components, TU Wien, Austria*; David Holec,* Department of Materials Science, Montanuniversität Leoben, Austria*; **Helmut Riedl [helmut.riedl@tuwien.ac.at]**,* Institute of Materials Science and Technology, TU Wien, Austria

Transition metal diborides, a class of refractory ceramics, have been shown to exhibit remarkable high-temperature stability and mechanical properties, encouraging research on their bulk and thin film forms. Scientific interest has been directed towards the formation of meta-stable solid solutions with silicon, with the aim of enhance oxidative properties and fracture characteristics. However, theoretical investigations of such ternary compounds remain rare. Therefore, in this study the structural, energetical, and mechanical properties of the Ti-Si-B_2, Zr-Si-B_2, and Hf-Si-B_2, as well as their vacancy dynamics, were explored with the help of Density Functional Theory (DFT). In all three systems, silicon is observed to prefer the boron sublattice. Through structural analysis, solubility limits of 24 at. %, 27 at. %, and 25 at. % of Si in $Ti(Si,B)_2$ $Zr(Si,B)_2$, and $Hf(Si,B)_2$, could be established, respectively. An analysis of simulated XRD patterns, Radial Distribution Functions (RDFs), and Crystal Orbital Hamilton Populations (COHPs), revealed that the loss of AlB_2-type symmetry could be attributed to the formation of Si clusters. Simulations of elastic properties demonstrated a reduction of Young's moduli but enhancing ductility criteria, both with increasing silicon contents, which was in line with experimental values up to 15 at. % Si. Concerning defects, the study revealed a structural instability of ternary AlB_2-type compounds with respect to metal vacancies. Furthermore, it was observed that both metal and boron vacancies showed a decreasing influence on the formation energies as the Si content increased.

MA-ThP-9 Fabrication and High-Temperature Test of Light-Weight Insulation Materials and Coatings for Reusable Thermal Protection Materials, ***Seongwon Kim [woods3@kicet.re.kr]**,* Korea Institute of Ceramic Engineering and Technology, Republic of Korea

Light-weight ceramic insulation materials and high-emissivity coatings were fabricated for reusable thermal protection systems (TPS). Alumina-silica fibers and boric acid were used to fabricate the insu¬lation, which was heat treated at 1250°C. High-emissivity coating of borosilicate glass modified with $TaSi_2$, $MoSi_2$, and SiB_6 was applied via dip-and-spray coating methods and heat-treated at 1100°C. Testing in a high-velocity oxygen fuel environment at temperatures over 1100°C for 120 seconds showed that the rigid structures withstood the flame robustly. The coating effectively infiltrated into the fibers, confirmed by scanning electron microscopy, energy-dispersive X-ray spectroscopy, and X-ray diffraction analyses. Although some oxidation of $TaSi_2$ occurred, thereby increasing the Ta_2O_5 and SiO_2 phases, no significant phase changes or performance degradation were observed. These results demonstrate the potential of these materials for reusable TPS applications in extreme thermal environments.

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