

## Protective and High-temperature Coatings

Room Palm 3-4 - Session MA3-2-TuA

### High Entropy and Other Multi-principal-element Materials II

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

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

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

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

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

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

**KEYWORDS:** (AlCrNbSiTiMo)N<sub>x</sub> high entropy alloy thin films, high power impulse magnetron sputtering, mechanical properties, corrosion resistance

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

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

In the second series, CrMoNbTiWC thin films were fabricated using the same Ti target power of 400 W and with varying carbon contents via a plasma emission monitoring (PEM) feedback control by a HiPIMS deposition system. With increasing carbon content, the CrMoNbTiWC thin film structure transitioned from BCC to FCC and then to amorphous. Wear tests revealed that the T80 film, containing 83.1 at.% carbon, exhibited the lowest friction coefficient (COF = 0.16) and a lower wear rate of  $2.1 \times 10^{-7}$  mm<sup>3</sup>/N·m, demonstrating excellent wear resistance. Corrosion tests showed that the T20 film with 36.5 at.% carbon had superior corrosion resistance in 0.5 M H<sub>2</sub>SO<sub>4</sub> (~1,500 times higher than 304SS), and in 3.5 wt.% NaCl (~33 times higher than 304SS). The detailed microstructure, mechanical, and corrosion properties of CrMoNbWTi and CrMoNbTiWC films with different Ti contents and varying carbon contents are comprehensively investigated in this study.

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

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

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

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

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

High-entropy alloys (HEAs) and high-entropy nitrides (HENs) have attracted increasing attention as protective coatings for demanding tribological and

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corrosive environments due to their high entropy, lattice distortion, and sluggish distortion effect. Compared to conventional transition-metal nitrides, HEN coatings can exhibit superior hardness–toughness, reduced wear, low friction coefficients, and enhanced corrosion resistance.

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

Nanoindentation showed hardness values ranging from ~17 to 31 GPa and reduced moduli between ~138 and 282 GPa, depending on film composition. Tribo-corrosion testing in 1 M H<sub>2</sub>SO<sub>4</sub> at open circuit potential (OCP) demonstrated that the most wear-resistant samples also exhibited the lowest coefficients of friction (~0.2), while sustaining the highest positive open circuit potentials (> 0.2 V) during sliding. Chromium-rich coatings with intermediate to low Si content, and reduced Ti and Zr concentrations outperformed the other compositions in terms of both wear and corrosion resistance. Wear volume analysis using optical profilometry revealed that all coatings significantly reduced wear compared to bare Ti6Al4V (by up to 260 times better), with the lowest wear rate of  $1.3 \times 10^{-6}$  mm<sup>3</sup>/N·m.

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

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

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