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

High Entropy and Other Multi-principal-element Materials III

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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