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

High Entropy and Other Multi-principal-element Materials I

Moderators: Erik Lewin, Uppsala University, Sweden, **Jean-François Pierson**, IJL - Université de Lorraine, France

8:40am **MA4-1-WeM-3 Growth and Properties of Epitaxial High-Entropy Alloy Thin Films*, Thomas Seyller (thomas.seyller@physik.tu-chemnitz.de),*** Chemnitz University of Technology, Germany **INVITED**

High-entropy alloys (HEAs) are discussed for applications in the fields of corrosion and wear protection as well as electrocatalysis. Although the surface properties of HEAs play a central role in these applications, they are still largely unexplored. This is - at least to a certain extend - caused by the unavailability of single-crystalline samples. In this presentation, recent progress is reported on the growth and subsequent characterization of epitaxial CoCrFeNi films [1]. The films were deposited by DC magnetron sputtering from spark-plasma sintered targets [2] using single-crystalline oxide substrates. A characterization of structural, chemical and electronic properties of the films was performed by different techniques including X-ray diffraction (XRD), scanning electron and transmission electron microscopy (SEM, TEM), energy-dispersive X-ray spectroscopy (EDX), X-ray photoelectron spectroscopy (XPS), angle-resolved photoelectron spectroscopy (ARPES), low-energy electron diffraction (LEED) and, more recently, by scanning tunnelling microscopy (STM). It is demonstrated that epitaxially grown HEA films have the potential to fill the sample gap, allowing for fundamental studies of properties of and processes on well-defined HEA surfaces over the full compositional space.

[1] H. Schwarz, J. Apell, H. K. Wong, P. Henning, R. Wonneberger, N. Rösch, T. Uhlig, F. Ospald, G. Wagner, A. Undisz, T. Seyller, Advanced Materials 35 (2023) 2301526 (https://doi.org/10.1002/adma.202301526).

[2] H. Schwarz, T. Uhlig, N. Rösch, T. Lindner, F. Ganss, O. Hellwig, T. Lampke, G. Wagner and T. Seyller, Coatings 11 (2021) 468 (https://doi.org/10.3390/coatings11040468).

9:20am **MA4-1-WeM-5 Effect of Elemental Additions (X: Pt, Al, Ti, and Ag) on the Microstructure and Electrical Properties of CrMnFeCoNiX-Based High-Entropy Alloy Thin Films*, Salah-eddine Benrazzouq (salah-eddine.benrazzouq@univ-lorraine.fr),*** *J. Ghanbaja, S. Migot, A. Nominé, J. Pierson, V. Milichko,* Institut Jean Lamour - Université de Lorraine, France

High-entropy alloys (HEAs) have garnered significant attention across various research and industrial fields owing to their exceptional properties, which originate from their complex multiprincipal element composition. This study delves into the phase evolution, microstructure, and electrical properties of the Cantor alloy (CrMnFeCoNi) enhanced by the incorporation of additional elements such as Pt, Ti, Al, and Ag. The deployment of DC magnetron co-sputtering has been crucial in achieving homogeneous films with precise stoichiometric and morphological control. This technique has enabled the systematic investigation of the structural evolution between crystalline phases (FCC, BCC) and amorphous states and their subsequent impact on the properties of the films. We carried out comprehensive characterization using X-ray diffraction (XRD), high-resolution transmission electron microscopy (HRTEM), scanning electron microscopy (SEM), resistivity measurements, and optical reflection measurements to assess the films' structural, microstructural, electrical, and optical attributes.

Abundant nanotwins were observed in the CrMnFeCoNi and CrMnFeCoNiPt films, both of which possessed a single FCC crystalline structure. The CrMnFeCoNiAl films transitioned from a single FCC phase to a duplex FCC + BCC phase structure, eventually stabilizing as a single BCC structure. The duplex FCC+BCC phase exhibited a low degree of nanotwins with larger grains of each phase. The CrMnFeCoNiTi films displayed an amorphous structure at various percentages, whereas the CrMnFeCoNiAg films exhibited a multiphase structure comprising single Ag and CrMnFeCoNiAg phases. Notably, Ag formed precipitates zone within the Cantor matrix. The observed phases were consistent with predictions made using thermodynamic criteria, despite the far-from-equilibrium conditions.

The study reveals that altering the concentration of elements such as Al and Pt significantly impacts the films' crystallographic structure and microstructure. Specifically, the electrical resistivity increased with the addition of elements in the single-phase regions. Notably, values of electrical resistivity were even higher in the duplex phase for the Al-doped samples due to the additional scattering effects of FCC/BCC phase boundaries in the alloys. The incorporation of silver was found to decrease the material's resistivity, likely because of the increased precipitation of silver within the Cantor matrix. Furthermore, optical reflectance and temperature-dependent electrical resistivity measurements confirm the metallic behavior of our alloys.

9:40am **MA4-1-WeM-6 Property Evaluation of Nd Doped NiCoFeAlTi Non-equiatomic High Entropy Alloy Films and the Influence of Post-annealing Treatment*, Chia-Lin Li (chialinli@mail.mcut.edu.tw),*** Center for Plasma and Thin Film Technologies, Ming Chi University of Technology, Taiwan

The effects of Nd addition on the microstructures and mechanical properties of non-equiatomic NiCoFeAlTi high entropy alloy films (HEAFs) were studied in this work. A series of NiCoFeAlTi HEAFs doped with Nd, ranging from 0 to 8.7 at.% Nd, was prepared by magnetron co-sputtering. Subsequently, a post-annealing treatment at 700°C was executed to investigate the changes in microstructure and mechanical properties exhibited by all films. The mechanical properties, phases and microstructures of Nd doped HEAFs and annealed films were characterized by the nanoindentation, X-ray diffractometer (XRD) and transmission electron microscopy (TEM), respectively. Based on XRD results, amorphous structures were identified in all Nd doped NiCoFeAlTi HEAFs. After annealing, the films exhibit a mixture of HEA FCC, NdNi HCP and L12 phases due to annealing-induced crystallization. For the mechanical properties of Nd doped HEAFs, both the hardness and elastic modulus showed an initial increase, reaching the maxima of ~9.3 GPa and ~158 GPa at 0.61 at.% Nd addition, respectively,and then decreased with the increasing Nd content. The influence of post-annealing treatment of Nd doped NiCoFeAlTi HEAFs on the microstructures and mechanical properties will be given further in this study.

11:20am **MA4-1-WeM-11 Effect of Substrate Temperature on Properties and Microstructure of High Entropy Alloy Thin Films Deposited by Magnetron Sputtering Systems*, Yi-Jun Yan (yjyan@gapp.nthu.edu.tw)*, *F. Ouyang,*** National Tsing Hua University, Taiwan

In recent years, high-entropy alloy thin films have attracted much attention because of their higher strength and lower cost than bulk materials. This study used magnetron sputtering to prepare $Ni_{30}Co_{30}Fe_{13}Cr_{15}Al_6Ti_6$ high-entropy alloy (HEA) thin film on a Si substrate. We investigated the effect of substrate temperature on the properties and microstructure of HEA thin films, including nanotwin formation, grain growth, hardness, and roughness. The composition of the film is uniformly distributed, and different substrate temperatures did not cause significant changes in the concentration of film elements. The thin film fabricated at low substrate temperature has a highly (111)-oriented columnar grain structure, and the nanotwin boundaries are parallel to the substrate surface with average twin spacing of 1.4 nm. As the substrate temperature increased, the columnar grain structure gradually disappeared and transformed into a BCC+FCC dual-phase polycrystalline structure. The hardness of thin film possesses a maximum hardness of 1.92 GPa at the substrate temperature of 100 ^oC, but as the substrate temperature rises, the grain growth and detwinning cause the hardness to decrease. The resistivity of HEAs is about 105 uΩ-cm, and there is no obvious correlation with the substrate temperature. The HEA thin films also exhibit a flat surface morphology with a root mean square roughness value of 0.5 nm at low substrate temperature. But the root mean square roughness value increased to 1.5 nm as substrate temperature increased, which is due to the grain growth inside the films. The residual stress of the film changed from compressive stress to tensile stress as the substrate temperature increases. The results of this study show that the substrate temperature greatly influences the microstructure, twin crystal growth, hardness, and residual stress, and corresponding mechanism will be discussed in the talk.

11:40am **MA4-1-WeM-12 A Combinatorial Approach to Developing Sputter-Deposited AuBiTaW High-Entropy Alloy Films for Inertial Confinement Fusion Applications*, Daniel Goodelman (goodelman1@llnl.gov)*,** *D. Strozzi, S. Kucheyev, L. Bayu Aji,* Lawrence Livermore National Laboratory, USA

After achieving inertial confinement fusion (ICF) ignition in December 2022, further optimization of material properties and experimental protocols are required to increase the fusion energy gain. To accomplish this goal, we are designing a new generation of hohlraums. Typically fabricated via magnetron sputtering, hohlraums are centimeter-scale sphero-cylindrical cans made from Au or depleted U with a wall thickness of >10 μm, serving as the outer housing for fusion fuel capsules. They must balance design constraints including high laser light-to-x-ray conversion efficiency, mechanical and corrosion stability, and electrical resistivity for magnetically assisted implosion. Here, we present results of a combinatorial magnetron co-sputtering study, aimed at developing a family of AuBiTaW films to address these outstanding challenges. Effects of the alloy composition and deposition process parameters on the microstructure, residual stress, mechanical properties, and electrical transport will be considered, as well as implications for ICF applications.

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.

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