Protective and High-temperature Coatings
Room Town & Country C - Session MA4-2-WeA

High Entropy and Other Multi-principal-element Materials II

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

2:00pm **MA4-2-WeA-1 Effect of Bilayer Periodic Thickness Ratios on the Mechanical Properties and Corrosion Resistance of TiZrNbTaFeN/TiN High Entropy Alloy Nitride Multilayer Thin Films***, **Sheng-Yuan Hung (jjj881029253@gmail.com)**,* Ming Chi University of Technology, New Taipei, Taiwan*; B. Lou,* Chang Gung University, Taoyuan, Taiwan*; J. Lee,* Ming Chi University of Technology, New Taipei, Taiwan

Due to the excellent mechanical and physical properties of high entropy alloy thin films, they have attracted extensive attention and research from the global industry, academia, and research institutions in recent years. In this study, an equimolar TiZrNbTaFe high entropy alloy target and Ti target were used to deposit TiZrNbTaFeN/TiN multilayer films on AISI304 stainless steel, AISI420 stainless steel, and silicon wafers substrates by a high power impulse magnetron sputtering (HiPIMS) system. The bilayer period thickness ratios of TiZrNbTaFeN and TiN layers were adjusted from 1:1 to 1:2 and 2:1. The cross-sectional morphology of each thin film was observed with a field emission scanning electron microscope. The crystal structure of the multilayered film was analyzed with an X-ray diffractometry. A nanoindenter, scratch tester, and pin-on-disk wear tester were used to measure the hardness, elastic modulus, adhesion, and wear resistance. The corrosion resistance of multilayered thin films in 0.1 M sulfuric acid aqueous solution was tested by the electrochemical workstation. Effect of bilayer periodic thickness ratios on the microstructure, mechanical properties, and corrosion resistance of TiZrNbTaFeN/TiN multilayer films will be explored

2:20pm **MA4-2-WeA-2 Enhanced Mechanical Properties of Nitrogen-Supersaturated High-Entropy Alloys via Phase Manipulation***, **Yujie Chen (yujie.chen@adelaide.edu.au)**,* University of Adelaide, Australia

N-supersaturated $Fe_{50}Mn_{30}Co_{10}Cr_{10}$ high-entropy alloys (HEAs) were prepared via magnetron sputtering at various N_2 flow rates (R_N) of 4, 8, 10, 15 and 20 sccm, denoted hereafter as N4, N8, N10, N15 and N20, respectively. It was found that the N content rose up from 6.5 to 28.9 at.% when R_N increased from 4 to 20 sccm. Both N4 and N8 exhibit a face-centred cubic (FCC) structure. An increase inR_N to 10 sccm and 15 sccm resulted in the formation of an FCC and hexagonal closed-packed (HCP) dual-phase structure. The volume fraction of the FCC phase increased with a further increase in R_N, leading to a predominant FCC structure in N20. Despite their unusually high N concentration of up to 28.9 at.%, the HEAs comprises solid solution phases without nitride formation. Notably, the N15 HEA with 21.8 at.% N shows an impressive hardness of 20 GPa, comparable to ceramics, while demonstrating exceptional damage-tolerance with considerable plasticity. The excellent combination of high hardness and damage-tolerance is believed to stem from 1) massive solid solution strengthening caused by a high level of N intake, 2) a dual-phase FCC and HCP structure supposedly due to the low stacking fault energy, and 3) stress-induced FCC to HCP phase transformation. These findings demonstrate that, in contrast to the high brittleness as seen in nitrides, N-supersaturated HEAs can undergo large plastic deformation like pure metallic materials, thus opening up a new avenue for enhancing the mechanical properties of advanced alloys for applications under extreme loading conditions.

2:40pm **MA4-2-WeA-3 Mechanical and Anticorrosive Properties of Laminated (NbTaMoW)N_x Films***, **Yan-Zhi Liao (11289034@mail.ntou.edu.tw)**, Y. Chen,* National Taiwan Ocean University, Taiwan

(NbTaMoW)N_x films were prepared through cosputtering with four element targets. The distinction in characterization between the laminated nitride films fabricated at substrate holder rotation speed R_H of 2 and 10 rpm and homogeneous high-entropy alloy nitride films prepared at R_H of 30 rpm were evaluated. The nitrogen flow rate (f_{N2} = [N_2/(N_2+Ar)]) during the sputtering process was set at 0.1, 0.2, and 0.4, respectively. The deposition rate decreased from 43.8 to 33.7 nm/min with increasing f_{N2} from 0.1 to 0.4 at R_H of 2 rpm due to the target poisoning effect, whereas the deposition rate decreased from 46.4 to 34.8 nm/min at R_H of 10 rpm. The phase structures and mechanical and anticorrosive properties of the (NbTaMoW)N_x films were studied. The results indicated that a metallic phase dominated structure was observed for the films prepared at f_{N2} of 0.1, whereas nanocrystalline and face-centered cubic nitride phases were obtained for films fabricated at f_{N2} of 0.2 and 0.4, respectively. The films deposited at f_{N2} of 0.4 exhibited hardness values of 25.2 and 26.1 GPa for the films prepared at R_H of 2 and 10 rpm, respectively, which were lower than 29.9 GPa for the films prepared at R_H of 30 rpm. Potentiodynamic polarization tests were conducted out for evaluating the anticorrosive properties of the films on SUS420 substrate.

3:00pm **MA4-2-WeA-4 Structure and Mechanical Properties of (Al,B,Cr,Si,Ti)-based Thin Films***, **Alexander Kirnbauer (alexander.kirnbauer@tuwien.ac.at)**, P. Konecny,* TU Wien, Institute of Materials Science and Technology, Austria*; R. Hahn,* Christian Doppler Laboratory for Surface Engineering of High-performance Components, TU Wien, Austria*; S. Kolozsvari,* Plansee Composite Materials GmbH, Germany*; P. Mayrhofer,* TU Wien, Institute of Materials Science and Technology, Austria

High-entropy alloys (HEAs) and high-entropy metal-sublattice ceramics (HESCs) have recently gained particular attraction in the field of materials research due to their promising properties, such as high hardness, high strength, and thermal stability. Ceramics based on the high-entropy concept mostly consist of refractory metals such as Ta, Hf, Zr, W, V etc. These metals are good nitride and carbide formers which is why they are mainly used especially for PVD coatings. Nevertheless, the production of these elements needs a lot of energy input due to their very high melting points. Furthermore, these elements are very heavy which in consequence makes them hard to process and rather expensive. In this study we want to focus on a material system consisting of Al, B, Cr, Si, and Ti which are comparably light and cheap elements and the production of a corresponding compound target consumes less energy. To get an idea of the properties of coatings based on this material system we investigated "metallic" coatings as well as nitrides and oxides. The coatings were synthesised by magnetron sputtering using a single composite target with an equiatomic composition and different gas mixtures. All the coatings produced show XRD amorphous diffraction patterns without any indication of crystalline phases. Also, SEM images of fracture cross-sections do not show the, usually characteristic, columnar growth which further underpins the results obtained by XRD measurements. The hardness and indentation modulus of the coatings range from ~10 to 22 GPa and from ~170 to 260 GPa, respectively, depending on the character of the coating. To get information of the bonding state, XPS measurements were carried out. Furthermore, in-situ cantilever bending tests were done to investigate the fracture toughness of the coating depending on their either "metallic", nitride, or oxide character.

3:20pm **MA4-2-WeA-5 Synthesis and Characterization of High Entropy Ceramic Coatings from Cr-Hf-Mo-Ta-W Refractory Metal System***, S. Debnárová, T. Stasiak, V. Buršíková,* Masaryk University, Czechia*; Z. Czigány, K. Balázsi,* HUN-REN Centre for Energy Research, Hungary*; S. Lin, N. Koutná,* Technische Universität Wien, Austria*; **Pavel Souček (soucek@physics.muni.cz)**,* Masaryk University, Czechia

High entropy alloys (HEAs) are multicomponent materials containing at least five principal elements with contents ranging between 5 and 35 at.%. The high entropy concept also extends to ceramics, such as oxides, nitrides, borides and carbides. High entropy materials can exhibit high strength and hardness at low as well as high temperatures, outstanding structural stability, wear, corrosion and oxidation resistance. This makes them promising candidates for next-generation replacements of traditional materials in many areas of the industry.

In this contribution, we are examining the formation of single-phase high entropy nitrides and high entropy carbides NaCl-type fcc structure from the Cr-Hf-Mo-Ta-W system. Magnetron sputtering was used for all depositions. An ambient temperature was used for the first deposition set, while an elevated temperature of 700°C was used for the second to observe the influence of the temperature on the crystallization. Argon/nitrogen gas admixture was used in nitrides, while argon/acetylene was used in carbides. This led to the first difference in reaching different nitrogen/carbon content in the coatings. While sputtering in nitrogen is a typical representative of reactive magnetron sputtering and nitrogen content never exceeded 50 at.%, sputtering in acetylene belongs to the hybrid PVD-PECVD deposition processes, also known as unsaturated reactive sputtering, and much higher carbon content in the coatings is reached. The deposition rate did not significantly decrease for all reactive gas flows. The structure and mechanical properties of the coatings were heavily influenced by the reactive gas flow for both systems. In films deposited without acetylene flow, a bcc metallic phase was observed.

Increasing reactive gas flow first showed an amorphous structure and then an fcc multielement carbide structure. Therefore, the ability of the system to form either metallic or ceramic nitride and carbide single phases was confirmed. Amorphous coatings exhibited a dense microstructure, while crystalline films were more columnar with multilayered structure at the nanoscale given by the deposition process geometry. The mechanical properties of the deposited films were good, exhibiting a hardness of up to 25 GPa, while the majority of the coatings were around 20 GPa. There was no great difference between the hardness of the corresponding nitrides and carbides.

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3:40pm **MA4-2-WeA-6 Mechanical and Oxidation Properties Evaluation of Equimolar and Non-Equimolar High Entropy Alloy Boron Carbonitride Coatings***, **Igamcha Moirangthem (igamcha@gmail.com)**,* National Taiwan University of Science and Technology, Taiwan*; B. Lou,* Chang Gung University, Taiwan*; C. Wang,* National Taiwan University of Science and Technology, Taiwan*; J. Lee,* Ming Chi University of Technology, Taiwan

In recent studies, high entropy alloy (HEA) nitride and carbide coatings have shown improved chemical and mechanical properties as compared to conventional alloy nitride and carbide coatings. Various combinations of transition metals in equimolar ratio as well as non-equimolar ratio carbide and nitride coatings have been explored recently for excellent mechanical and chemical properties using physical vapor depositions. In this study, an equimolar TiZrNbTaFe alloy target and a non-equimolar $Al_4Cr_2NbSiTi_2$ alloy target were used to fabricate their boron carbonitride phases using superimposed high power impulse magnetron sputtering. A radio frequency (RF) power source was used for the boron target. The coatings were deposited on p-type Si (100), AISI 304, and AISI 420 stainless steel substrates. The nitrogen flow was maintained at a constant rate, and the acetylene flow rate was varied. The microstructures, phases, and surface roughness of the HEA boron carbonitride coatings were investigated by a field emission scanning electron microscope, X-ray diffractometer, and atomic force microscope, respectively. The nanohardness was measured using nanoindentation. A pin-on-disk tribometer was used to study the wear characteristics of these coatings. The effects of heat treatment, oxidation, and potentiodynamic polarization of these coatings were also examined. The mechanical, chemical, and oxidation properties of TiZrNbTaFeBCN and AlCrNbSiTiBCN boron carbonitride coatings were explored in this work.

4:00pm **MA4-2-WeA-7 Research on the Effects of Various Acetylene Contents on the Mechanical Properties of TiZrNbTaFeBCN High Entropy Alloy Films***, **Meng-Hsueh Chuang (norman12lin@gmail.com)**,* National Taiwan University of Science and Technology, Taiwan*; B. Lou,* Chang Gung University, Taiwan*; J. Lee,* Ming Chi University of Technology, Taiwan*; C. Wang,* National Taiwan University of Science and Technology, Taiwan

The conventional alloys are made of one primary element with the addition of small or moderate amounts of alloying elements to yield an alloy with specific properties. However, in 2004, Professor Jien-Wei Yeh from National Tsing Hua University, Taiwan, and Professor Brain Cantor from the University of Oxford, UK, independently introduced innovative material systems known as multicomponent alloys and high entropy alloys (HEAs). These breakthroughs garnered significant attention and research interest from the global academic, industrial, and scientific communities and led to a new distinct branch in materials research. The effects of various acetylene gas flow rates on the chemical composition, microstructure, phase structure, hardness, and wear resistance of TiZrNbTaFeBCN high entropy alloy films were investigated. The HEA thin films were prepared by co-sputtering an equimolar TiZrNbTaFe high entropy target and a TiB_2 target onto the surfaces of AISI420 stainless steel, AISI304 stainless steel, and P-type (100) silicon wafer substrates using fixed nitrogen gas flow rate and different acetylene flow rates. The structures of thin films were determined by an X-ray diffractometer. The cross-sectional morphologies of thin films were examined by field emission scanning electron microscopy (FE-SEM). A nanoindenter and scratch test were used to evaluate the hardness and adhesion properties of thin films, respectively. Effects of carbon contents on the mechanical properties of TiZrNbTaFeBCN HEA thin films will be discussed.

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