Advanced Surface Engineering
Room Central Exhibit Hall - Session SE-ThP
Advanced Surface Engineering Poster Session

SE-ThP-1 High-Temperature Oxidation Resistance of Sputtered (Al,Cr,Nb,Ta,Ti,Si)N Coatings*, Andreas Kretschmer,* TU Wien, Austria*; P. Mayrhofer,* TU Wien, Institute of Materials Science and Technology, Austria

High-entropy metal-sublattice (Al,Cr,Nb,Ta,Ti)N coatings, with up to 15.0 at.% additional Si content were developed and investigated for their oxidation resistance by exposing them for 3, 30, and 100 h to ambient air at 900, 950, and 1000 °C, which represents the harshest oxidation experiment for crystalline nitrides reported so far. The Si-free coating is rapidly oxidized, but all of the Si-alloyed coatings survive even the harshest oxidation test. The oxides crystallize mostly in the rutile structure with some Ta2O5-type phase fractions at higher Si contents. Detailed TEM investigations reveal a varied microstructure across the oxide scales with a succession of Cr-, Al, and Ti-rich top oxide layers, which agrees with a reported thermodynamical calculation of oxide stabilities.

SE-ThP-2 ASED Rising Star Talk: The Influence of Deposition Parameters on the Structure and Properties of TiZrNbTaMo High Entropy Alloy Films Fabricated by High Power Impulse Magnetron Sputtering*, Chia-Lin Li*[1], Center for Plasma and Thin Film Technologies, Ming Chi University of Technology*; S. Hou,* Department of Materials Science and Engineering, National Tsing Hua University*; B. Lou,* Chemistry Division, Center for General Education, Chang Gung University*; J. Lee,* Department of Materials Engineering, Ming Chi University of Technology*; P. Chen,* Department of Materials Science and Engineering, National Tsing Hua University, Taiwan

TiZrNbTaMo high-entropy alloys (HEAs) with a body-centered cubic (BCC) structure are known for their excellent compressive yield strength and significant compressive plasticity. These advantageous properties are retained even in their thin film forms, making them highly promising for a variety of applications. In this study, we prepared TiZrNbTaMo high entropy alloy films (HEAFs) grown by high power impulse magnetron sputtering and investigated the influence of deposition parameters on their structure and properties. It is well known that several deposition parameters affect the density and microstructure of thin films, influencing the hardness, wear resistance, toughness, and corrosion resistance. The cross-sectional morphology and crystal structure of TiZrNbTaMo HEAFs were characterized using field emission scanning electron microscope (FE-SEM) and X-ray diffractometry (XRD), respectively. Mechanical properties of the HEAFs, including hardness, elastic modulus, adhesion, and wear resistance, were evaluated by nanoindentation, scratch tester, and pin-on-disk wear tester. This study systematically investigated the effects of critical processing parameters, including pulse frequency, duty cycle, substrate bias, and working pressure for TiZrNbTaMo HEAFs for achieving outstanding properties.

SE-ThP-3 Enhanced SiO_2/Ag Multilayer Coatings via Magnetron Sputtering: Advancing Potential Applications in Removable Implants*, Magali Restrepo Posada,* Universidad de Antioquia, Colombia

The success of soft metal-doped ceramic coatings in biomedical applications can be attributed to their ability to provide optimal mechanical strength and bactericidal properties in removable implants. However, a primary challenge associated with these devices is the colonization of bacteria on the implant surface, leading to potential infections and inflammation in surrounding tissues. To mitigate these issues, the exploration of SiO_2 coatings has been proposed due to their proven mechanical strength, thermal stability, and biocompatibility. Despite these advantages, SiO_2 coatings may not effectively combat all bacterial strains, limiting their applicability in the implant industry. Thus, incorporating antibacterial materials such as silver (Ag) is recommended to enhance the coatings' antibacterial efficacy.

The fabrication process involved magnetron sputtering, resulting in the formation of a SiO_2/Ag multilayer system characterized by high homogeneity, strong adhesion, and compactness. The surface and cross-sectional morphology of the coatings were assessed using field emission scanning electron microscopy (FESEM), while contact angle and roughness were measured. Mechanical properties were evaluated through nanoindentation, and chemical and structural analysis were conducted using X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), and scanning electron microscopy with energy-dispersive spectroscopy (SEM/EDS).

SE-ThP-4 Investigating the Atomistics underlying Magnetron Sputter Deposition of Mo Thin Films and their Microstructure/Property Influence*, Joyce Custer,* Sandia National Laboratories

Multimodal materials datasets provide the large amount of information needed for expediting the discovery of process-structure-property relationships important to materials performance. We describe a multimodal dataset for sputter-deposited molybdenum thin films. The dataset is for 27 unique depositions that vary sputter power and pressure.

The atomistics of deposition are investigated using the binary-collision Monte Carlo computer program SIMTRA. This program simulates the gas-phase transport of sputtered species tracking the collisions of sputtered metal atoms with argon (Ar) process gas. SIMTRA outputs the energy and angular distributions of incident metal species for the various process conditions. The SIMTRA simulations account for the complex sample rotation associated with true planetary stage motion. The predicted kinetic energies and incidence angles are reported using both probability density functions and uniformity wafer maps.

Grain structure and crystal texture were investigated using cross section transmission electron microscopy and X-ray diffraction. High angle annular dark field and bright field cross-section transmission electron micrographs were obtained from films produced in each of the depositions. Automated crystal orientation mapping was used to derive inverse pole figures from the imaged areas covering hundreds of grains, and MTEX, a Matlab toolbox for analyzing crystallographic textures, extracted statistics of the grain sizes and tilt. Analysis shows the emergence of tilted columnar grains and branched grain structure with increasing sputter pressure. These changes are correlated to reduced metal atom kinetic energy and broad distributions of incidence angles (predicted by SIMTRA).

The atomistics of deposition are also correlated with keyphysical properties including film density and residual stress. Increased hyperthermal energies generally lead to denser thin films consistent with TEM observations. Films follow previously reported trends of compressive in-plane stress at low process pressure transitioning to tensile or near-zero stress at elevated values. Using the multimodal dataset, we attempt to correlate several key stress signatures (e.g., compressive-to-tensile transition and peak stress) to atomistic details to provide insight into the relative roles of average kinetic energy and average incidence angle.

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SE-ThP-5 Magnetron Sputtering Deposition of Metallic-Based Nanostructured Coatings for Nuclear Energy Applications*, Maria Sole Galli De Magistris, D. Vavassori, V. Russo, D. Dellasega,* Politecnico di Milano, Italy*; M. Gentile, F. Garcia Ferré,* newcleo Srl, Italy*; M. Passoni,* Politecnico di Milano, Italy

Lead-cooled Fast Reactors (LFRs) are considered among the most promising Generation IV nuclear reactors owing to their inherent safety and high power density [1]. Nevertheless, conventional structural and cladding materials suffer from severe corrosion issues when in contact with liquid lead [2]. Surface coating technology is a valuable technique to improve materials performance in harsh environments without modifying the properties of the bulk [3]. Nonetheless, coatings must also withstand the high temperature and radiation fields characterizing LFRs. Therefore, realizing coatings with improved adhesion and precisely controlled properties is of fundamental importance. In this respect, in recent years, metallic coatings have gained increasing interest thanks to their enhanced compatibility with steel substrates and the possibility of triggering a self-passivation mechanism when strong oxide formers, such as Al and Cr, are present in the optimal amount. Physical Vapor Deposition techniques, particularly Magnetron Sputtering (MS) [4], have proven their effectiveness in realizing films with controlled and tunable properties. Magnetron Sputtering includes different deposition regimes: Direct Current (DCMS), Radio-Frequency (RFMS), and High Power Impulse Magnetron Sputtering (HiPIMS). Compared to DCMS and RFMS, the HiPIMS process generates a higher fraction of ionized species, with energies further adjustable through substrate biasing or Bipolar HiPIMS. Additionally, according to the working regime, a plethora of process parameters can be adjusted, including sputtering power, duty cycle, pulse width, and bias voltage amplitude, enabling film optimization for the specific application.

This contribution reports the development and characterization of advanced nanostructured mono and multi-elemental metallic coatings

[1] **Rising Star**

produced via magnetron sputtering. In particular, the attention is focused on two types of coatings. The first set consists in steel films, enriched in other elements such as aluminum and tungsten to improve the oxidation and the high-temperature behavior. The other set consists of chromium films; widely investigated for claddings in light water reactors, they might be of interest for LFRs owing to their excellent oxidation behavior. Different deposition conditions were exploited and relevant working parameters were adjusted to tune coatings properties. Morphological and crystallographic studies were performed, together with preliminary tests in LFR relevant conditions. The obtained results provide an insight into the interconnection between process parameters and coatings properties and behavior.

SE-ThP-6 UV-Vis-NIR Optical Analysis to Understand the Electrical Properties of Nitrogen-Incorporated Tetrahedral Amorphous Carbon Thin Films, ***Nina Baule***, Fraunhofer USA Center Midwest; *D. Tsu,* The Mackinac Technology Company; *L. Haubold,* Fraunhofer USA Center Midwest; *T. Schuelke,* Fraunhofer USA

Tetrahedral amorphous carbon (ta-C) thin films have received significant attention due to their diamond-like mechanical properties, achieved via low-temperature synthesis. More recently, the electrochemical behavior of nitrogen-incorporated ta-C:N has attracted interest, as it exhibits electrical conductivity as well as chemical inertness. The modified electrical properties of ta-C:N have been mainly attributed to the conjugation of sp^2 carbon-carbon and/or carbon-nitrogen bonds. Commonly, with an increase in nitrogen incorporation into the growing film, the electrical resistivity decreases proportionally. Here, however, we find that the electrical properties plateau at higher nitrogen content for ta-C:N thin films (100 nm) deposited by laser controlled pulsed cathodic vacuum arc (Laser-Arc), despite the fact that mechanical and structural properties indicate an increase in sp^2 fraction. Hence, to gain a better understanding of the electronic properties, the dielectric constants [ε_1] and [ε_2] were obtained from the optical constants, the refractive index [n] and extinction coefficient [k], which were numerically determined from reflectance [R] and transmittance [T] measurements in the wavelength range from 190 to 2500 nm (0.5 to 6.5 eV). The dielectric constant [ε_1] was used to calculate the dielectric volume of the atom, which is interpreted as a measure of the conjugated electron system. Furthermore, modeling of the dielectric constants yielded the number of conjugated electrons per atom. The ta-C:N samples with the highest values of electrical conductivity, were either characterized by the largest occupied volume or the highest number of conjugated electrons. Through the dielectric volume and number of conjugated electrons, it was discovered that the electrical conductivity does not only depend on the sp^2 content and cluster size, but on how many electrons are conjugated and how much space these electrons occupy.

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