Advanced Surface Engineering
Room Central 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-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 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.

SE-ThP-7 A Critical Issue in Coatings Nanoindentation: Validity of the 10% Bückle's Rule of Thumb*, **Esteban Broitman,*** SKF Research and Technology Development, Netherlands

When an indenter penetrates the surface of a film deposited onto a substrate, the mechanical response of the coating will be influenced by the mechanical properties of the substrate, according to its penetration depth h and the film thickness t. As the depth of penetration h increases, more of the mechanical contribution will come from the substrate.

The first who tried to separate the contribution of the substrate from the total measured hardness at the microscale was Bückle, who suggested a 10% rule of thumb: to indent no more than 1/10 of the film thickness to avoid the influence from the substrate. The rule has been adopted later by many researchers for nanoindentation experiments and extended also as valid for the elastic modulus. However, there are many experimental studies and numerical simulations showing that this rule is too strict for a hard coating on a very soft substrate and too loose for a soft coating on a hard substrate [1].

In this presentation, we will review the issue, and will discuss all factors that affect the maximum penetration depth for independent coating measurements. We will also present a simple experimental methodology

[1] **AVS Rising Star**

that, in most of cases, gives the correct values for hardness and elastic modulus, independently of the coating/substrate system.

[1] E. Broitman, Indentation Hardness Measurements at Macro-, Micro-, and Nanoscale: A Critical Overview. Tribol Lett **65**, 23 (2017)

SE-ThP-8 Optimizing Laser Surface Melting Parameters to Enhance Corrosion Resistance of AA5083 Aluminum Alloy*, **Md Sojib Hossain**,* University of Virginia, USA, Bangladesh*; W. P. Moffat,* University of Virginia, USA*; J. Skelton,* University of Virginia, USA, United States Virgin Islands*; J. Fitz-Gerald,* University of Virginia, USA

AA5083 is a solution-strengthened Al-Mg alloy widely used in marine environments and other harsh, corrosive settings due to its excellent corrosion resistance. However, this alloy is susceptible to a deleterious process known as sensitization, where Mg-rich intermetallic particles, primarily the β-phase, precipitate at grain boundaries under prolonged exposure to moderately elevated temperatures. This anodic β-phase forms a galvanic couple with the cathodic aluminum matrix, leading to intergranular corrosion (IGC). Although several heat treatment processes have been developed to dissolve the β-phase within the matrix, these methods are not feasible for service components, and processed samples can easily be re-sensitized. Laser surface melting (LSM) offers a promising solution to this issue. In this study, a nano-second pulsed laser with varying power was used to investigate LSM's effects on minimizing intergranular corrosion. A low-power excimer laser with 50% overlap was applied to a 1200-grit polished surface, while a high-power ADAPT laser was used on as-received samples with different overlaps. Electrochemical polarization scans of the base and LSM samples in a 0.6 ml NaCl solution showed notable improvements in corrosion properties for all laser-processed samples, as evidenced by low open circuit potential and nearly a two-order-of-magnitude decrease in exchange current density. Backscattered scanning electron micrographs and energy dispersive spectroscopy (EDS) scans confirmed that the improved corrosion resistance was due to a homogenized LSM surface, ranging from only 3 to 7 µm in depth. Electron backscattered diffraction (EBSD) analysis of the excimer LSM samples' cross-sections identified very small sub-grains with different grain boundary characteristics compared to the sensitized base metal. These sub-grains are highly inert to re-sensitization, and create significant interruptions to intergranular corrosion, thereby enhancing IGC resistance.

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