Advanced Surface Engineering
Room 125 - Session SE-MoM

Plasma-Assisted Surface Modification and Deposition Processes/Nanostructured and Multifunctional Coatings

Moderators: Diana Berman, University of North Texas, **Filippo Mangolini**, The University of Texas at Austin

8:15am **SE-MoM-1 Materials Design in Surface Engineering***, **Johanna Rosen,*** Linköping University, IFM, Sweden **INVITED**

MAX phases are a family of atomically laminated ceramics where M is a transition metal, A is a group A element, and X is C or N. These materials are a playground for design of both three- and two-dimensional (3D/2D) phases, for diverse applications. MAX phases are to date primarily synthesized in powder form, but we present epitaxial thin films of Ti_3AlC_2 and Ti_3SiC_2 on sapphire through magnetron sputtering from three elemental targets. We show that Ti_3AlC_2 can be converted to 2D Ti_3C_2 MXene through selective etching of Al in hydrofloric acid (HF), while Ti_3SiC_2 can be transformed into 3D Ti_3AuC_2, $Ti_3Au_2C_2$ and Ti_3IrC_2 by noble metal substitution reaction, the latter forming high-temperature-stable Ohmic contacts to SiC. Evidence is also presented for synthesis of single-atom thick layers of gold by selective removal of Ti_3C_2 from Ti_3AuC_2 by Murakami's reagent. Insight into these 3D and 2D materials and the methods by which they are formed is given through a combination of first principles simulations and electron microscopy, which suggest additional pathways for design of new phases.

8:45am **SE-MoM-3 Development of Texture in Ta_2C Thin Films Sputter-Deposited on Free-Standing Graphene***, **Suneel Kodambaka,*** Virginia Tech*; K. Tanaka,* University of Chicago

Thin crystalline films are commonly deposited on bulk solids and the development of texture (preferred orientation) in such thin films is reasonably well understood.[1] Efforts to grow highly crystalline thin films have included, for example, the use of low-energy ion irradiation[2] and van der Waals (vdW) epitaxy.[3] Exciting developments based on vdW epitaxy include the use of vdW buffer layers (e.g., graphene, and hBN) on crystalline substrates to grow highly oriented MoS_2 and $(VNbTaMoW)S_2$ thin films.[4, 5] Recently, Koichi *et al*.[6] reported that Ta_2C thin films sputter-deposited on hBN/Ta_2C surfaces are more highly oriented than those grown using the same deposition parameters on bare Ta_2C. These results led us to question the need for a bulk substrate and if crystalline thin films can be directly deposited on *free-standing* vdW layers instead.

Here, we present results obtained from sputter-deposition of Ta_2C films on monolayer-thick graphene substrates and on relatively thicker (~8 nm) amorphous silicon nitride (a-SiN_x) membranes supported by transmission electron microscopy (TEM) grids. Using plan-view TEM and selected area electron diffraction, we compare and contrast the microstructures of the Ta_2C films on graphene and a-SiN_x. We find that the Ta_2C layers deposited on a-SiN_x are composed of a mixture of nanoscale crystallites and non-crystalline phases, while the Ta_2C film on graphene is polycrystalline with grains that are oriented in-plane as $[2\text{-}1\text{-}10]_{film}||\ [10\text{-}10]_{graphene}$. These results indicate that even a single-atom-thick crystal can promote crystalline and oriented growth.

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9:00am **SE-MoM-4 Manufacture and Microstructure of Tantalum Nitride Films by Radio Frequency and High Power Impulse Magnetrom Sputtering Techniques***, Y. Chiang, Y. Chang, **Fan-Bean Wu,*** National United University, Taiwan

Tantalum nitride, TaN, film attracted attension for decades due to its merits in thermal, mechanical, tribological, electrical properties and had been employed in versatile applications, including microelectronics, seiconductor, protective layer and so on. However, the TaN films frequently deposited through sputtering process in vacuum possessed various microstructural features according to deposition conditions, leading to the evolution in characteristics. A comparative study focused on the control on fabrication parameters of inlet gas ratio, input power types, including radio frequency, and high power impulse magnetron sputtering, i.e. RFMS and HiPIMS, respectively, and the duty cycle modulation of the HiPIMS technique was conducted. An amorphous/nanocrystalline microstructure feature could be deduced under a low RF power, while a higher level of RF power enhanced the crystallization of the TaN films. The even higher power density upto 0.5 kW by the HiPIMS technique triggered a multiphase microstructure comprised of TaN, Ta2N, and TaN2 phases. Under such high power density, a strong columnar feature was obtained regardless of the duty cycle. In addition, under a higher Ar/N_2 gas ratio of 18/2 with limited nitrogen the TaN showed a stoichiometry of Ta2N, while an elemental ratio Ta:N=1:1 was achieved with a ratio of Ar/N_2=15/5. Recent findings on microstructure evolution and related characteristics of the TaN coatings were discussed.

9:15am **SE-MoM-5 In-Situ Laser Diagnostics of Plasma Surface Interactions by Fs-TALIF***, **Mruthunjaya Uddi,*** Advanced Cooling Technologies*; G. Urdaneta, A. Dogariu,* Texas A&M University

Plasma surface interaction has been a critical area of research for many applications such as Plasma-Enhanced Atomic Layer Etching (PEALE). To meet the demanding needs of more advanced atomically controlled microfabrication methods, the physics of PEALE needs to be better understood to enable high quality, repeatable and controllable deposition process. Several challenges that need to be addressed regarding PEALE include damage to the substrate from highly energetic species and UV radiation, need for precise amorphous/crystalline modulated selective layer deposition, conformality in coating non-uniform substrates, achieving an aspect ratio of >100, repeatability and controllability of the finish. To address these challenges, we are developing laser diagnostics methods to measure species over substrates by advanced laser diagnostics such as femtosecond- Two-Photon Absorption Laser Induced Fluorescence (fs-TALIF) to image atomic species over substrates. Here we present measurements of N, O atom densities over a substrate with high spatial (< 10 microns) and temporal resolution (<1 ns) using fs-TALIF at pressures of 5-150 mTorr. Temperature was measured over a substrate surface using NO 2-line LIF using femtosecond laser excitation.

9:30am **SE-MoM-6 Interlayer Optimization for Nitrogen-Incorporated Tetrahedral Amorphous Carbon Thin Film Optically Transparent Electrode***, **Nina Baule**, D. Galstyan, L. Haubold,* Fraunhofer USA Center Midwest

While several studies around the usage of nitrogen-incorporated tetrahedral amorphous carbon (ta-C:N) in electroanalysis have been published, they mainly focus on ta-C:N films deposited on conductive substrates. This is due to the relatively high resistivity of ta-C:N compared to other carbon and metal-based electrodes: without the electrically conducting substrate, there are high ohmic losses in the ta-C:N when subjected to an electrical current. This limits the use of ta-C:N in optically transparent electrodes (OTEs), which must be deposited on optically transparent (typically electrically insulating) substrates such as quartz. In this study we deposited 50 nm of ta-C:N by laser controlled pulsed cathodic vacuum arc (Laser-Arc) onto insulating quartz substrates to investigate the electrochemical response compared to the same film deposited on conductive silicon. To test the responses of the films, we performed electrochemical oxidation/reduction of potassium ferrocyanide during cyclic voltammetry (CV). Here we find that no oxidation or reduction during CV could be observed at the ta-C:N electrode deposited on quartz. To address this and to maintain optical transparency over the visible wavelength range, we then introduced a 5 nm chromium (Cr) interlayer deposited by magnetron sputtering between the ta-C:N and quartz. While this electrode configuration led to clear cathodic and anodic CV peaks of potassium ferrocyanide, the peak separation compared to the ta-C:N deposited on conductive silicon was increased. That finding indicates that the electrode has a higher resistance. However, we further improved ta-C:N's electrode functionality on quartz by optimizing the Cr sputtering

conditions and introducing a plasma pretreatment by a single-beam ion source. Atomic force microscopy revealed that these changes caused an improved Cr growth homogeneity, which led to the enhanced electrical conductivity. These results show that ta-C:N's potential as an OTE is not precluded by its high ohmic losses on insulating substrates. In fact, the promise of mechanically stable and electrochemically active ta-C:N requires only that a conductive interlayer be used, and these films could impact the realms of optical materials, flexible electronics, sensors, and more.

9:45am **SE-MoM-7 Highly-Ordered Metallic Nanostructure Arrays: Strategies, Status, and Challenges***, **Jinn P. Chu**,* National Taiwan University of Science and Technology, Taiwan

This presentation reports on the wafer-scale fabrication of metallic nanostructure arrays with highly ordered periodicity. With the semiconductor-based lithography and sputter deposition, various metallic arrays including metallic nanotube array, metal mesh, and metallic pillar array are fabricated. The array structure is manufactured by sputtering metals onto a contact-hole array template created in the photoresist by photolithography. Following sputter deposition, the photoresist and any excess top-layer coating were removed using acetone, leaving behind the nanotube array on the substrate. The efforts were recognized with the American Chemical Society (ACS) Award at the nano tech 2018 International Nanotechnology Exhibition & Conference in Tokyo, Japan. We utilized both ferrous (stainless steel) and nonferrous alloys (Cu-, Ni-, Al-, and Ti-based), elemental metals (Cu, Ag, and Au), as well as various oxides to form these array structures. The proposed arrays can be fabricated over a wide range of heights and diameters (from a few hundred nm to 20 μm) and in various shapes, including tall cylinders, dishes, triangles and rhombuses. Furthermore, when combined with other nanomaterials (e.g., ZnO nanowires, graphene oxide, or Au nanoparticles), arrays become nanohybrids suitable for many applications. These applications include thermal emitters, triboelectric nanogenerators, SERS-active biosensors, and anti-icing devices.

10:00am **SE-MoM-8 Refining Deposition and Thermal Processes for High-Quality Bi-Mo-O Thin Films***, R. Gonzalez-Campuzano, A. Hernandez-Gordillo, **Sandra Elizabeth Rodil**,* Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México

Bismuth molybdates, a family of Bi-Mo-O materials with diverse elemental compositions and crystalline structures, have been extensively investigated for their excellent catalytic properties in the oxidation of lower olefins. These properties facilitate the synthesis of organic chemicals widely used in the plastic industry. Their photocatalytic activity under visible light has recently been demonstrated in micro and nano powder samples. However, there has been limited research on the deposition and characterization of Bi-Mo-O materials as thin films. Since 2016, Matova et al. have shown that sputtering deposition using Bi and Mo targets in an $Ar+O_2$ atmosphere is feasible, demonstrating the visible-light photocatalytic degradation of dyes in water. Despite these promising results, the broader application of this bismuth-based photocatalyst has seen limited advancement. To address this gap, our research group has renewed the investigation of Bi-Mo-O semiconductors for photocatalytic degradation of recalcitrant pollutants, leveraging their active response to visible light. In this study, we report on the thin-film deposition of Bi-Mo-O samples using a co-sputtering system with Bi_2O_3 and Mo targets. The direct current power applied to the Mo target varied from 20 to 60 W, while the RF power applied to the Bi_2O_3 target was fixed at 30 W, allowing us to achieve films with different Bi/Mo ratios. The substrate was pre-heated to 420 K and rotated at approximately 10 RPM to ensure film uniformity. Our results indicated that the films transitioned from crystalline to amorphous as the Mo content increased. Annealing experiments using rapid thermal processing equipment, introducing air into the chamber, performed at 773.15, 873.15, and 973.15 K for 20 minutes for each sample, aiming to obtain various Bi-Mo-O crystalline structures. The structure variations, optical band gap, and bonding-composition analysis are presented. Mo-rich samples presented high optical absorption with band gaps below 0.5 eV, but most samples presented band gaps in the visible-range. Pure phase Bi-Mo-O films and heterostructures containing MoO_x phases were obtained and tested for the degradation of the indigo carmine dye under visible light.

10:30am **SE-MoM-10 Low-Temperature Synthesis of Stress-Free, Ceramics Thin Films Using Metal-Ion Irradiation***, **Ivan Petrov**,* Univerisity of Illinois at Urbana-Champaign*; L. Hultman, G. Greczynski,* Linköping University, IFM, Sweden

Ion irradiation is a key tool for controlling epitaxy-to-nanostructure, phase content, and physical properties of refractory ceramic thin films grown by magnetron sputtering. Until recently, thin film growth relied on enhancing adatom mobility in the surface region by inert and/or reactive gas ion irradiation to obtain dense layers at low deposition temperatures. Development of high-power pulsed magnetron sputtering (HiPIMS), which provides metal-ion plasmas with tunable degree of ionization, enabled systematic studies of the effects of metal-ion irradiation on properties of refractory ceramic thin films. A motivation for the use of metal-ions stems is that they are film constituents, hence they can provide the benefits of ion-mixing without causing the high compressive stresses associated with trapping of gas ions at interstitial sites.

This presentation reviews growth experiments of transition metal nitride model systems including TiAlN, TiSiN, VAlN, TiTaN, TiAlTaN, and TiAlWN. Film synthesis is carried out in a hybrid configuration with one target powered by HiPIMS and other operated in direct current magnetron sputtering (DCMS) mode.A substrate bias potential V_s is synchronized with the metal-ion-rich portion of the HiPIMS pulses to control the metal-ion energy. The time-resolved mass spectrometry analyses performed at the substrate position enables us to suppress the role of gas ion irradiation and select intense

Irradiation with lower-mass metal-ions (Al^+ or Si^+) results in near-surface implantation with the depth controlled by V_s amplitude. This enables synthesis of *metastable* ternary cubic Me_1Me_2N solid solutions far above the Me_1N concentration range achieved by DCMS. At the other end, bombardment of the growing film surface with pulsed high-mass metal ion fluxes (W^+ or Ta^+)during hybrid HiPIMS/DCMS high-rate deposition of dilute $Ti_{1-x}Ta_xN$, $Ti_{1-x-y}Al_xTa_yN$, and $Ti_{1-x-y}Al_xW_yN$ alloys provides high fluxes of low energy recoils and results in fully-dense, low-stress, hard and superhard coatings without external substrate heating (temperature ≤130 °C).

10:45am **SE-MoM-11 ASED Young Investigator Award Finalist Talk: Understanding Ceramics Under Extreme Mechanical Loads via Machine-Learning Potential Molecular Dynamics***, **Nikola Koutna**[1], S. Lin,* TU Wien, Austria*; L. Hultman, D. Sangiovanni,* Linköping Univ., IFM, Thin Film Physics Div., Sweden*; P. Mayrhofer,* TU Wien, Austria

Inherent brittleness and easy crack formation are serious challenges for applications of hard ceramic films. Prior to the development and targeted testing of a specific material, data-driven ab initio and machine-learning techniques can facilitate efficient and relatively inexpensive screening of the relevant chemical space with desired structure–property constraints. Furthermore, theoretical approaches can aid experiment in providing atomic-to-nanoscale understanding of deformation and crack initiation processes under well-defined loading conditions. In this talk I will discuss the exciting and rapidly growing field of machine-learning interatomic potentials (MLIPs) for molecular dynamics and how these can be used to study boron-based ceramics under extreme mechanical loads, highly relevant for applications of these materials. Transition metal diborides (TiB_2, TaB_2, WB_2) and MAB phases (nanolaminates alternating ceramic-like, Ti–B, Ta–B, W–B, and metallic-like, Al, layers) will be used to exemplify a possible MLIP training strategy as well as to discuss challenges upon up-scaling beyond ab initio length scales. Uniaxial tensile tests as well as compression tests with a surface pre-crack will be simulated for supercells with up to 10^6 atoms, previously inaccessible to both ab initio as well as molecular dynamics calculations due to the size limitations (ab initio) and due to the fact that only few interatomic potentials for ceramics exist (molecular dynamics) and basically none has been properly tested for large-scale simulations including severe mechanical loads. Equipped with the newly developed machine-learning interatomic potentials, I will further discuss strain-induced nucleation of extended defects MAB phases and relate them to relevant experimental observations.

11:00am **SE-MoM-12 ASED Rising Star Talk: Coupling CdS/g-C_3N_4 Heterojunctions with Remarkably Transfers Process: Impact of Stacking Grade of g-C_3N_4 Micro-flakes***, **Karen Valencia García**[2], A. Henández Gordillo, S. Rodil Posada,* National Autonomous University of Mexico

In this work, heterojunction materials of cadmium sulfide (CdS) with carbon nitride (g-C_3N_4) were prepared and the photocatalytic activity in hydrogen (H_2) production was studied using an ethanol-water solution. The influence of ammonia (NH_3) on the physicochemical and photocatalytic properties of g-C_3N_4 was investigated. It was evaluated in the photocatalytic H_2 production, obtaining a null response, but the g-C_3N_4 exhibit activity in the photodegradation of the indigo carmine dye (*IC*) solution using blue LED light. From the analysis of the results, a parameter defined as

[1] **ASED Young Investigator Award Finalist**
[2] **Rising Star**

SA/(WCA*gap) (**surface area** (SA); **water contact angle** (WCA) and photon absorption (**band gap**)) is proposed to show how the different **surface parameters** in the photocatalytic response. Subsequently, the effect of the amount of g-C_3N_4 on the heterojunction formed with the CdS nanofibers, which were synthesized in the solvent mixture of ethylenediamine and butanol, was studied. The heterojunction of CdS/g-C_3N_4 was carried out using the g-C_3N_4 synthesized by polymerization with 1 mL of hydrazine (UH1, the g-C_3N_4 with the maximum value of **SA/(WCA*gap**) and with the g-C_3N_4 synthesized with 2 mL of hydrazine (UH2, the g-C_3N_4 that presented the greatest physical-chemical and optoelectronic modification). The CdS heterojunctions with the modified g-C_3N_4 exhibited a high H_2 production rate of (5258 μmol $h^{-1}g^{-1}$) ~2.0 times higher than the unmodified CdS nanofibers. The increase in H_2 production rates of the heterojunctions was related to the coupling of the CdS nanofibers on the surface of g-C_3N_4 lamellar plate: (1) result of a better capacity to absorb visible light; (2) the lower resistance to charge transfer, decreasing the recombination of the e^-/h^+ pairs. For the heterojunctions, the increase in photocatalytic activity suggests that the coupling of the CdS materials with g-C_3N_4 was satisfactorily achieved, observing a synergy of CdS with g-C_3N_4. Physical mixtures equivalent to heterounions were made, and they presented a low rate in the evolution of H_2, the low activity is due to the fact that there is no coupling between the CdS nanofibers and g-C_3N_4.

Keywords: heterojunctions, hydrogen production, photocatalysis.

11:15am **SE-MoM-13 ASED Young Investigator Award Finalist Talk: Advanced EMI Shielding with Quantum Dots and 2D Nanomaterial Enhanced Dual-Polymer Fiber Films**, ***Lihua Lou*[1]**, Florida International University; *G. Al-Duhni,* Florida International University, Jordan; *O. Cruz,* Florida International University, Nicaragua; *J. Volakis, M. Pulugurtha, A. Agarwal,* Florida International University

An ultra-thin, lightweight, and highly flexible nanocomposite film is developed by synergistically integrating iron oxide quantum dots (FeQDs) and graphene nanoplatelets (GNPs), specifically targeting electromagnetic interference (EMI) shielding applications. To enhance the electrical conductivity of the resulting thin film, a dual-faceted strategy is employed: utilizing a hybrid polymer system as the matrix and constructing a QDs/2D nanomaterial-integrated multilayer network within the film's architecture. This intricate design approach facilitates a robust investigation into the fiber-based thin films' structural, chemical properties, electrical conductivity, and EMI shielding capabilities, including characterization and simulation methodologies. Findings reveal that the electrospun fibers of 10GNP-1QDs exhibit an average diameter of ~613 ± 192 nm, presenting a significantly higher surface roughness than the pristine PAN fibers. This morphological variance is attributed to the intricate particle-polymer interactions. Raman spectroscopy analysis confirms the successful incorporation of GNPs and FeQDs into the fiber matrix, as evidenced by slight shifts in peak positions, indicative of atomic and molecular interactions between the composite's organic and inorganic constituents. Electrical conductivity measurements underscore a remarkable figure of 350,000 S/m, a characteristic partially ascribed to GNPs and FeQDs' facilitative role in enhancing the polymer matrix's conductive pathways. The magnetic SE within the frequency range of 250 to 1000 MHz spans between 30 to 35 dB, surpassing the performance of all other thin films, including control samples fabricated through coating and casting methodologies. This enhanced performance is linked to the improved electron mobility afforded by FeQDs. Additionally, within the low-frequency range of 0 to 1 MHz, the film exhibits an SE ranging from 40 to 50 dB, markedly outperforming Al and Cu films of equivalent thickness. Notably, within the high-frequency X-band spectrum of 8 to 12 GHz, the SE reaches levels up to 170 dB, ~30 dB higher than that of Al and Cu films. Furthermore, across the far-field frequency range of 100 MHz to 12 GHz, the film demonstrates an SE between 65 to 100 dB. The predominant shielding mechanisms contributing to these outcomes include absorption, multi-reflection, reflection, hysteresis loss, and polarization loss, collectively ensuring the nanocomposite's superior performance in EMI shielding applications. This exploration significantly advances the field by demonstrating the exceptional capabilities of 1D/2D nanomaterial-integrated thin films across a wide frequency spectrum.

11:30am **SE-MoM-14 ASED Young Investigator Award Finalist Talk: Functionalizing GaN Surfaces for Enhanced Soft Tissue Integration in Biomedical Implants**, ***Monu Mishra*[2]**, Dyal Singh College, University of Delhi, India; *J. Sharan,* All India Institute of Medical Sciences, Bhuvneshwar, India; *M. Kashyap,* Jawaharlal Nehru University, India; *G. Gupta,* CSIR National Physical Laboratory, India

Biomedical implants are a routine treatment serving the recovery of functional difficulties in human beings to live a better quality of life. Implants play a vital role in rebuilding and healing the damaged parts of human body including orthopaedic (bone support/replacement) and dental (maxillofacial reconstruction) related challenges. Implant surfaces having the first interaction with human body, thus govern the quality of implant-tissue integration leading to success/failure and the life of the subjected implant. The commercially available titanium based biomedical implants (i.e., Ti-6Al-4V) also suffer and are often failed due to the poor integration of soft tissues with implant surfaces. In recent years, Gallium Nitride (GaN) being a biocompatible, aqueous and chemical stable material has emerged as a promising biomaterial offering enormous opportunities to modulate the surface chemistry to promote adhesion of targeted biomolecules for its efficient utilization in biomedical implants. Thereby, here we report a new three step process for the functionalization of GaN surfaces to promote the attachment/growth of human periodontal ligament fibroblast cells and reduce soft tissue integration related failures in biomedical implants. We have modified the morphology and surface chemistry of GaN thin films using sodium hydroxide (NaOH) and 3-aminopropyltriethoxy silane (APTES) followed by its bioconjugation with type 1-human collagen (T1HC). The changes in chemical bonding, morphology, wettability, pH, and aqueous stability of surface functionalized films have been investigated after every step using XPS, SEM and Contact Angle Measurement. Finally, the cell culture studies analysing cell proliferation and adhesion/survivability studies at different time intervals have been performed using MTT Assay and SEM evaluation. The studies divulge that chemically functionalized T1HC bio-conjugated GaN surfaces display excellent cell culture properties with a cell viability of ~ 95%. It demonstrates that GaN films can be useful in various procedures where integrating soft tissues with biomedical implant is highly desirable.

[1] **ASED Young Investigator Award Finalist**

[2] **ASED Young Investigator Award Finalist**

Advanced Surface Engineering
Room 125 - Session SE-MoA

Surface Engineering Solutions for Sustainable Development

Moderators: Ivan Petrov, University of Illinois at Urbana-Champaign, **Fan-Bean Wu**, National United University, Taiwan

1:30pm **SE-MoA-1 Towards Responsible Surface Engineering Based on PVD Technology**, ***Marcus Hans***, *J. Schneider,* RWTH Aachen University, Germany*; A. Matthews,* The University of Manchester, UK*; C. Mitterer,* Montanuniversität Leoben, Austria **INVITED**

The sustainable development goal (SDG) 12 'Responsible Consumption and Production' of the United Nations deals with the necessary change of consumption and production patterns towards a more sustainable future. Plasma-assisted physical vapor deposition (PVD) is increasingly employed to address global challenges, such as energy efficiency and reduction of CO_2 emissions. Two important questions are critically evaluated in this context:

1) How sustainable are state-of-the-art PVD processes and materials?

2) Which pathways are needed for future responsible surface engineering?

While our modern world and human life benefit from surface engineering, the consideration of energy and mass balances demonstrates that state-of-the-art PVD processes and products are not necessarily sustainable, leaving space for innovation. Responsible surface engineering comprises pathways to enhance the sustainability of processes as well as materials. Impurities will be discussed with respect to tolerable levels and even exploitation of 'impurities', which can be beneficial for the performance of a coating. Moreover, the microstructural design of chemically simple coatings offers opportunities to avoid economically and ecologically expensive elements. Prospective product cycles of emerging technologies and future products will enable the evaluation of ecological, economical as well as societal costs and benefits. Finally, responsible surface engineering involves a change in mindset of materials scientists, process engineers and of all stakeholders involved in garnering innovation.

2:00pm **SE-MoA-3 ASED Rising Star Talk: Unprecedented B Solubility in Cubic (Hf,Ta,Ti,v,Zr)B-C-N Coatings**, ***Andreas Kretschmer***[1], TU Wien, Austria*; A. Kirnbauer,* TU Wien, Institute of Materials Science and Technology, Austria*; R. Frost, D. Primetzhofer,* Uppsala University, Sweden*; M. Hans, J. Schneider,* RWTH Aachen University, Germany*; P. Mayrhofer,* TU Wien, Institute of Materials Science and Technology, Austria

In the past, we have studied the system (Hf,Ta,Ti,V,Zr)B-N with exceptional hardness and thermal stability, but the coatings contained a significant amount of C impurities, which may have influenced the properties [1]. To investigate the impact of C in this system, we have deposited new coatings with a TiN target, on which we placed diboride and/or carbide pieces of the metals Hf, Ta, V, and Zr. We have varied the composition by using either only diborides, only carbides, or different mixtures of the two material types to make 5 coatings containing either N and B, N and C, or all three. The B concentration varies between 42 and 0 at%, the C content between 25 and 0 at%, and the N content is stable at around 30 at% in all coatings. The Ti makes up roughly 20 at%, while the other metals are in the range between 2 and 5 at%. X-ray diffraction (XRD) shows a weakly textured single-phase fcc solid solution in all coatings. The FWHM of the 200 reflex ranges from 2 ° in the C-free coating down to 0.5 ° in the B-free coating, indicating different grain sizes. This is confirmed by transmission electron microscopy, revealing fine columnar growth in the 2.3 to 3.2 µm thick coatings, with especially fine grains in the B-richer coatings. Electron diffraction confirms that no secondary phases are present. We annealed the coatings in a vacuum furnace at 1000, 1200, and 1400 °C for 10 min, followed by XRD and nanoindentation. The coatings stay stable up to 1200 °C and start decomposing at 1400 °C. The as-deposited hardness of all coatings lies between 36 and 38 GPa, and is maintained after annealing at 1000 °C. After annealing at 1200 °C, the coatings containing only C or only B both soften to ~34 GPa, while the coatings with both C and B do not lose any hardness at this temperature. Only after annealing at 1400 °C does the hardness of all coatings drop below 30 GPa. We confirmed the thermal stability by atom probe tomography on the most promising sample, hereby we show that despite the high B content of 22 at%, decomposition into a diboride phase initiates only after annealing at 1400 °C. We further investigated the fracture-toughness by in-situ micromechanical cantilever bending. The best performing coating yields 4.0±0.5 MPa*m^(1/2), thus surpassing other similar coatings not only in hardness and thermal stability, but also fracture toughness.

[1] Kretschmer, A., et al. (2022). *Materials & Design*, *218*, 110695. https://doi.org/10.1016/j.matdes.2022.110695

2:15pm **SE-MoA-4 ASED Rising Star Talk: High Temperature Behavior of $Ti_{0.12}Al_{0.21}B_{0.67}$ Coatings Investigated by High-Resolution Transmission Electron Microscopy and DFT Calculations**, ***Sebastian Lellig***[2], RWTH Aachen University, Germany, Switzerland*; A. Navidi Kashani,* RWTH Aachen University, Germany*; P. Schweizer,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland*; M. Hans, G. Nayak,* RWTH Aachen University, Germany*; J. Michler,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland*; J. Schneider,* RWTH Aachen University, Germany

The oxidation behavior of stoichiometric $Ti_{0.12}Al_{0.21}B_{0.67}$ coatings is investigated by high resolution Transmission Electron Microscopy (TEM) after oxidizing for 1, 4 and 8 h at 700 °C and at 800 and 900 °C.

In the as deposited state, a ~ 4 nm thick, native, amorphous oxide layer covers the surface of the coating while the magnitude of incorporated O along the column boundaries decreases with depth. During oxidation, the formation of scale layers consisting predominantly of Al, O and B is observed that appear to be amorphous at 700 °C in which, after oxidation at 900 °C for 8h, nanocrystalline $Al_5(BO_3)O_6$ regions form. Concurrently, underneath the scale, the formation of Al- and Ti-rich boride regions, consistent with spinodal decomposition, is observed. Chemical environment dependent DFT predictions of the energies required for mass transport on the metal sublattice indicate that that Al diffusion is initiated before Ti. Hence, as the temperature is increased, the migration of Al is initiated first, leading to the formation of the oxide scale observed already after oxidation at 700 °C after 1 h. Below the oxidized region, the formation of Al-rich and Ti-rich regions by spinodal decomposition require the concurrent migration of Al and Ti. The fact that decomposition takes place at 900 °C and hence at larger temperatures than the Al diffusion mediate scale formation is consistent with DFT predictions as the averages of the predicted energies required for both, vacancy formation and migration for Ti are larger than for Al.

2:30pm **SE-MoA-5 Surface Engineering for New Highly Graphitized Carbon with High Pyrrolic-N as a Robust Support for Pt Electrocatalysts**, *H. Lee, D. Lee,* ***Jong-Sung Yu***, DGIST, Republic of Korea

For polymer electrolyte membrane fuel cells (PEMFCs), the state-of-the-art electrocatalysts are based on carbon-supported Pt group metals. However, current carbon supports suffer from carbon corrosion during repeated start-stop operations, causing performance degradation. We report a new strategy to produce highly graphitized carbon with controllable N-doping that uses extremely low-temperature synthesis (650 ℃) from g-C_3N_4 carbon-nitrogen precursor with novel surface engineering pyrolysis using Mg. The high graphiticity is confirmed by high-intensity 2D Raman peak with low I_D/I_G (0.57), pronounced graphitic XRD planes, and excellent conductivity. Without further post-treatment, this robust highly graphitized N-doped carbon (HGNC) material combines high pyrrolic-N content with high porosity.

Supporting Pt on HGNC exhibits excellent oxygen reduction activity for PEMFC with greatly improved durability as proved by real-time loss measurements of Pt and carbon, the first to surpass the DOE 2025 durability targets for both catalyst and support. The Pt/HGNC prepared at 650 ºC shows 32 and 24% drops in mass activity after accelerated durability tests of both electrocatalyst and support, respectively, which are less than DOE target of 40% loss. These values represent extremely small drop compared to those of commercial Pt/C, underscoring the merits of the novel low-temperature surface engineering pyrolysis with Mg. The atomistic basis for this durability is explained via quantum mechanics-based molecular dynamics simulations. Interestingly, it is found that pyrrolic-N strongly interacts with Pt, making the Pt catalyst more stable during fuel cell reaction. We expect that high porosity robust graphitized materials will lead to many other novel applications for electrocatalysis with durable carbon frameworks.

[1] **Rising Star**

[2] **Rising Star**

2:45pm **SE-MoA-6 ASED Rising Star Talk: Tunable Tribochemical Behavior of Pt-Au Thin Film Alloys Using High-Throughput Testing**, ***Tomas Babuska***[1], *F. DelRio, J. Hall, B. Boyce, D. Adams, J. Custer, M. Jain,* Sandia National Laboratories*; J. Killgore,* NIST-Boulder*; F. Mangolini, C. Edwards,* University of Texas at Austin*; J. Curry,* Sandia National Laboratories

Binary alloy systems such as Pt-Au have been shown to exhibit ultra-low wear and high hardness enabled by its intrinsically thermally stable nanocrystalline microstructure making them suitable candidates for electrical contact materials. When the unique mechanical properties are combined with the catalytic behavior of platinum under cyclic shearing in inert environments, adventitious carbon can be mechano-chemically transformed into lubricious surface films ($\mu < 0.05$). While ideal for solid lubrication applications, carbon deposits can inhibit electrified interfaces and compromise end-use performance. By balancing the catalytic and mechanical properties of Pt-Au thin films, the friction behavior and surface film morphology can be controlled to either promote/prevent lubricious carbon film formation.In this work, we explore 448 compositions of sputter deposited Pt and Au thin films spanning 0-100 at% and the intertwined role of hardness and composition on the resulting friction and wear behavior. We highlight the use of custom high-throughput tribological platforms with robotic automation to rapidly test large material spaces and for the creation of in-depth structure-property relationships that will play a crucial role in the development of next-generation tribological coatings. SNL is managed and operated by NTESS under DOE NNSA contract DE-NA0003525.

3:00pm **SE-MoA-7 ASED Rising Star Talk: Advanced Hipims Nanocrystalline and Metallic-Glass High Z Coatings for Interaction with Liquid Metals**, ***Davide Vavassori***[2], *L. Bana, M. Bugatti, M. Galli De Magistris,* Politecnico di Milano, Italy*; M. Iafrati,* Department of Fusion and Technology for Nuclear Safety and Security, ENEA, Italy*; D. Dellasega, M. Passoni,* Politecnico di Milano, Italy

Liquid metals (LMs) are of interest for the development of several applications related to the energy sector. However, the presence of LMs creates concerns about the compatibility with standard structural materials since, generally, LMs affects their integrity and mechanical properties through different phenomena [1]. Therefore, the mitigation of LM corrosion emerges as a crucial step. This especially applies if the aim is the integration of LMs in a nuclear energy system which, being characterized by high temperatures and strong radiation fields, introduces constraints on the choice of possible structural materials.

The deposition of nanocrystalline or metallic-glass protective coating is an appealing approach to engineer the structural material surface and, consequently, to control the interaction with LMs. In this respect, the fine tailoring of the coating properties at the nanoscale represents a key aspect to optimize its performances in the harsh LM environment. To this end, the use of an of advanced Physical Vapor Deposition methods such as High Power Impulse Magnetron Sputtering (HiPIMS) [2] can play a key role to produce films with controlled properties.

In this framework, a notable example is represented by the magnetic confinement fusion research area where innovative divertor designs based on liquid tin (Sn) are under investigation [3,4]. Nevertheless, the action of liquid Sn in detrimental for many structural materials typically used in fusion systems. Specifically, liquid Sn heavily corrodes the heath-sink components which are constituted by CuCrZr alloy to satisfy the thermomechanical and cooling requirements imposed by the fusion power flux.

This works aims at investigating the corrosion resistance of tungsten (W)-based coatings produced by HiPIMS when in contact with liquid Sn. To this end, two different strategies have been studied to grow the metallic coating on CuCrZr substrates. On the one hand, the application of different pulsed substrate bias voltages synchronized with the HiPIMS pulse were considered to produce pure W coatings. On the other hand, the co-sputtering from a W target and an aluminium (Al) target, both working in HiPIMS mode, was examined to realize a multilayer coating. The produced samples were tested in corrosion experiments carried out at 400 °C for relatively short periods of time (150 or 600 minutes). The characterization of the samples before and after the exposure allowed to evaluate how coatings properties determined their effectiveness as protective layer and, thus, to retrieve preliminary information about their ability to withstand the typical operation condition expected in a LM-based divertor.

3:15pm **SE-MoA-8 Eliminating Surface Charging in X-Ray Photoelectron Spectroscopy of Insulators for Reliable Bonding Assignments**, ***Grzegorz (Greg) Greczynski***, Linkoping University, Sweden

Sample charging during X-ray photoelectron spectroscopy (XPS) analyses of electrically insulating samples is a widely recognized challenge of this essential technique. If the electron loss caused by the photoelectric effect is not compensated due to specimens' poor electrical conductivity, the positive charge building up in the surface region results in an uncontrolled shift of detected core level peaks to higher binding energy (BE). This seriously complicates chemical bonding assignment, which is based on measured peak positions, and accounts for a large spread in reported core level BE values. Here, we show that peaks from several industry-relevant oxides, serving as model insulators, typically displaced by several eV due to charging, shift back to positions characteristic of electrically-neutral samples following *ex-situ* capping with a few nm thick metallic layer with low affinity to oxygen. The effect is present only if the capping layers contain sufficiently large non-oxidized volume that provides long-range conduction paths to grounded Cu clamps, while being thin enough to allow for recording high quality spectra from the underlying insulators. The versatility of the charging elimination is demonstrated for different oxides/cap combinations and air exposure times. The method is robust and easy to apply.

3:30pm **SE-MoA-9 ASED Rising Star Talk: Friction and Wear of MXene/MoS2 Nanocomposite Coating Under Dry and Hydrocarbon-Lubricated Conditions**, ***Ali Zayaan Macknojia***[3], *A. Voevodin, S. Aouadi,* University of North Texas*; S. Berkebile,* Army Research Laboratory*; D. Berman,* University of North Texas

Friction and wear-related failures remain the greatest problems in moving mechanical assemblies operating under various conditions. This study demonstrate lubricity achieved by spray-coating solution-processed multilayer Ti3C2Tx-MoS2 blends onto rough 52100-grade steel surfaces. Blends exhibited lower frictional performance for individual pristine materials, MoS_2 and $Ti_3C_2T_x$, under high pressure, sliding speed. Study investigated the processing, structure, and property correlation to gain a deeper understanding of the underlying phenomena. Raman spectroscopy, scanning electron microscopy, and transmission electron microscopy results revealed the formation of an in-situ robust tribolayer responsible for the outstanding performance observed at high contact pressures and sliding speeds. This study has broad implications for the development of solid lubricants that can operate under extreme conditions and low viscosity fuel environment, inspiring further research and development in this field.

4:00pm **SE-MoA-11 STrengthening Mechanisms for High Entropy Alloy Coatings Fabricated by Magnetron Sputtering**, ***Jyh-Wei Lee***, Ming Chi University of Technology, Taiwan, Republic of China*; B. Lou,* Chang Gung University, Taiwan

Prof. Yeh has been developing high entropy alloys (HEAs) for twenty years.The research on bulk HEA materials has attracted much attention due to their unique properties being better than those of traditional alloys. On the other hand, the HEA coatings fabricated by magnetron sputtering methods have been ensured for improving substrate materials' mechanical properties, corrosion resistance, oxidation resistance, and wear resistance. In this work, several strengthening mechanisms, including nitridation, carburization, solid solution hardening, and grain refinement,were adopted to study their effects on the mechanical properties improvements of VNbMoTaW, VNbMoTaWAl, VNbMoTaWCr, TiZrNbTaFe, TiZrNbTaFeB, and ZrTiNbSiFe high entropy alloy coatings grown by magnetron sputtering technique. The chemical compositions, phase structures, microstructures, and surface roughness of these HEA coatings were examined. The nanohardness, reduced elastic modulus, and wear resistance of HEA coatings were measured by nanoindenter and pin-on-disk wear tester, respectively.

We can conclude that good mechanical properties, including higher hardness and lower wear rate, can be obtained for those HEA coatings through the proper selection of strengthening mechanisms and the addition of several constituents, such as nitrogen, carbon, TiB_2, and Cr elements. This work evaluated some strengthening mechanisms and promising results of HEA coatings that can be used as protective coatings in harsh environments.

[1] **Rising Star**

[2] **Rising Star**

[3] **Rising Star**

4:15pm **SE-MoA-12 ASED Rising Star Talk: Multifunctional Optical Surfaces for Displays: From Antireflective to Self-Cleaning and Antimicrobial Functionalities**, ***Iliyan Karadzhov***[1], *C. Graham, A. Mezzadrelli,* ICFO-Institut de Ciencies Fotoniques, Spain*; W. Senaratne, K. Koch, P. Mazumder,* Corning Research and Development Corporation*; V. Pruneri,* ICFO-Institut de Ciencies Fotoniques, Spain

Glass is an indispensable part of display technologies, and knowing how to incorporate multiple useful properties into a single optical surface will enhance their performance. For example, nature-inspired designs can deliver high transparency and clarity, broadband and omnidirectional optical response, self-cleaning capabilities, and mechanical resistance. However, fabricating these surfaces with the desired properties can be complex, often requiring multistep lithography methods, which are costly and not easy to scale. In this talk, we discuss our team's recent developments in utilizing thermal dewetting of ultrathin metal films (Cu, Ag, Ni) as a lithography-free method to create durable, nanostructured optical surfaces with tailored multifunctional features.

In the first part, we demonstrate a transparent anti-microbial coating on glass surface based on dewetted copper (Cu) nanoparticles encapsulated by conformal SiO_2 and fluorosilane functional layers. The coatings kill more than 99.9% of *Staphylococcus aureus* within 2 hours due to the released Cu ions and can maintain their anti-microbial properties after wiping tests. The relatively flat transmission of 70-80% in the 380-750 nm range along with color neutrality and non-conductivity make them suitable for high-touch surfaces in medical and public settings where hygiene is important.

The second part focuses on a simplified method to create abrasion-resistant antireflective glass surfaces by creating randomly arranged subwavelength nanoholes. The fabrication process involves three main steps. First, silver nanoparticles are obtained by quickly thermally annealing an ultra-thin silver film on the glass substrate. These particles then serve as a base for a secondary etch mask, created by depositing a thin nickel layer over the silver nanoparticles and performing selective chemical wet etching. Finally, this mask is used in a dry etching process to carve nanoholes of varying depths into the glass surfaces. We achieve a transmission above 99% across a broad wavelength range with minimal scattering, where maximum spectral performance can be tuned to either the visible or near-infrared range by adjusting the lateral arrangement of the silver nanoparticles and the depths of the nanoholes. After undergoing an abrasion test of 10,000 passes with cheesecloth under constant load, the nanoholes remain structurally intact due to the redistribution of shearing mechanical forces.

4:30pm **SE-MoA-13 Effect of Europium and Gadolinium Alloying Elements on the Tribological Response of Low Hydrogen Content Amorphous Carbon**, *C. Edwards, H. Lien, N. Molina,* ***Filippo Mangolini,*** The University of Texas at Austin

Dopants and alloying elements are commonly introduced in amorphous carbon (a-C) materials to tailor their mechanical and tribological properties. While most published studies have focused on doping and/or alloying a-C coatings with metals or metalloids, doping a-C films with rare-earth elements has only recently been explored. Notably, our understanding of the shear-induced structural changes occurring in rare-earth element-containing a-C films is still elusive, even in the absence of any liquid lubricants. Here, the friction response of Eu- and Gd-containing a-C films with low hydrogen content deposited by high-power impulse magnetron sputtering (HiPIMS) on silicon was evaluated in open air and at room temperature. The load-dependent friction measurements indicated that the introduction of Gd ((2.3 ± 0.1) at.%) and Eu ((2.4 ± 0.1) at.%) into the a-C matrix results in a significant reduction of the shear strength of the sliding interfaces ((41 ± 2) MPa for a-C, (16 ± 1) MPa for a-C:Gd2.3 at.%, and (11 ± 2) MPa for a-C:Eu2.4 at.%.). Near-edge X-ray absorption fine structure (NEXAFS) spectromicroscopy experiments provided evidence that no stress-assisted sp3-to-sp2 rehybridization of carbon atoms was induced by the sliding process in the near-surface region of undoped a-C, while the amount of sp2-bonded carbon progressively increased in a-C:Gd2.3 at.% and a-C:Eu2.4 at.% upon increasing the applied normal load in tribological tests. The formation of an sp2-bonded carbon-rich surface layer in a-C:Gd2.3 at.% and a-C:Eu2.4 at.% films was not only proposed to be the origin for the reduced duration of the running-in period in tribological tests, but was also postulated to induce shear localization within the sp2-carbon rich layer and the transfer film formed on the countersurface, thus decreasing the interfacial shear strength. These findings open the path for the use of Gd- and Eu-containing a-C even under critical conditions for nearly hydrogen-free a-C films (i.e., humid air).

[1] **Rising Star**

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.

Sandia National Laboratories is managed and operated by NTESS under DOE NNSA contract DE-NA0003525.

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 [$\varepsilon_{2.}$] 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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