## 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.

**References**


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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**

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