Advanced Surface Engineering
Room 205 ABCD W - Session SE-TuA

Smart Coatings and Responsive Surfaces: Engineering for Tomorrow

Moderator: Filippo Mangolini, The University of Texas at Austin

4:00pm **SE-TuA-8 Spatial Configurations in Magnetron Sputtering: A Comprehensive Review**, ***Esteban Broitman***, *Rickmer Kose,* SENTYS Inc.*; Sven Kelling,* SENTYS, Inc.

Magnetron sputtering stands at the forefront of thin-film deposition, with its efficacy intimately tied to how magnetron sources and substrates are arranged in space. In this review, we distill experimental results from across the literature into three core configurational categories: sputter-up versus sputter-down magnetron orientations, planar versus confocal magnetron arrays, and on-axis versus off-axis substrate alignments.

For each configuration, we explore how geometry shapes plasma confinement, steers the angular distribution of sputtered atoms, and dictates film characteristics—density, residual stress, microstructural evolution, and step coverage. By correlating specific geometric features with these critical film properties, we introduce a decision framework that guides researchers in selecting the optimal magnetron arrangement to achieve a targeted material performance.

To illustrate practical implementation, we showcase contemporary deposition chambers and magnetron source designs engineered for rapid, tool-free adjustment of magnetron–substrate geometry, empowering users to fine-tune film growth in situ.

4:15pm **SE-TuA-9 Wide-Bandgap Hybrid Metamaterials: Theory guided Advanced Surface Engineering for UV active Photonic Properties**, ***Ufuk Kilic***[12], *Shawn Wimer, Matthew Hilfiker, Raymond Smith,* University of Nebraska-Lincoln*; Christos Argyropoulos,* The Pennsylvania State University*; Eva Schubert, Mathias Schubert,* University of Nebraska-Lincoln

Metamaterials (MMs) -the artificially engineered surface structures with subwavelength scale features- are at the forefront of optoelectronic, quantum, and biomedical advancements [1-4]. Despite the critical importance, their effective operation in the ultraviolet (UV) spectral range by using wide-bandgap materials (WBGM) for aforementioned advancements is seldom discussed in the literature [1]. WBGMs provide exceptional transparency, high stability, corrosion resistance, and UV-active optical responses. These properties enable strong UV-active light-matter interactions, making them ideal for robust, tunable MMs in advanced photonic and quantum applications.

In this study, our methodology is framed over a theory-guided approach for fabricating and optimizing MM platforms from ultra-wide bandgap Zirconia (ZrO_2). While the finite element modeling provides insights on light-matter interaction at nanoscale [2-4], Monte Carlo ballistic simulation method unravels the particle flux dynamics and the structure growth process [5]. Utilizing electron beam assisted glancing angle deposition technique, that is particularly known for its capacity to produce various 3D morphologies over wafer-scale area, and free of masks [2-4], we fabricated highly ordered nano-columnar, and nano-helical MM platforms. Using Mueller Matrix generalized spectroscopic ellipsometry technique, we optically investigated the fabricated MM platforms within the spectral range covers near-IR (0.64 eV) to vacuum-UV (9.5 eV) and found that they exhibit strong optical anisotropies including circular dichroism and birefringence.

Here, we also present and discuss the subsequent depositions of dielectric (ZrO_2) and metallic (silver/Ag) materials leading to hybrid plasmonic MMs with a multiple number of subsegments that achieve enhanced and spectrally controlled optical anisotropies active in visible to UV spectral range. Performing complementary scanning electron microscopy, transmission electron microscopy, and energy-dispersive X-ray spectroscopy, we extracted the integrity, crystallinity, and stoichiometry of the fabricated MM platforms. This work advances photonic and quantum device design by integrating material fabrication, theoretical modeling, and experimental characterization, demonstrating how wide-bandgap ZrO_2 combined with plasmonic metals enables tunable MMs for high-power systems, UV photonic circuits, and chiral sensors.

[1]Duncan, M. A.,et al.,ACS Appl.Mater.Interfaces,14(50),55745-55752,(2022)

[2]Kilic, U.,et al.,Adv.Funct.Mater.31.20:2010329,(2021)

[3]Kilic, U.,et al.,Adv.Opt.Mat.2302767,(2024)

[4]Kilic, U.,et al.,Nat.Comm.15.1:3757,(2024)

[5]Wimer, S.,et al.,Vacuum,(under review 2025)

4:30pm **SE-TuA-10 On the Energy Efficiency of Sputtering of Elemental Targets by Inert Gas Ions Ne, Ar, Kr, and Xe**, ***Ivan Petrov,*** University of Illinois at Urbana Champaign*; Michal Fečík, Stanislav Mráz, Jochen Schneider,* RWTH Aachen University, Germany

Environmentally responsible surface engineering has emerged as an important topic in academic and industrial research. A recent review article[1] provides an extensive overview of sustainability aspects of physical vapor deposition (PVD) processes, focusing on magnetron sputtering and cathodic arc deposition. The authors point out that “energy and mass balances are an important sustainability-relevant aspect, constituting tremendous untapped potential for the surface engineering community”. Sputtering by particle bombardment produces energetic species which contribute to low-temperature growth of high-quality coatings and films. A large portion of the incoming energy is, however, converted to heat in the targets. Therefore, it is of interest to optimize the energy and the mass of the inert gas to make sputtering more energy efficient.Here we attempt to quantify the fraction of the incoming energy which is transferred to the sputtered atoms for elemental targets as a function of ion energy for four inert gases, Ne, Ar, Kr, and Xe. Previously, Carter et al[2] introduced the term erosion efficiency, $\eta^{er} = U.Y(E)/E$, where U is the sublimation energy of the metal target. This definition includes the potential energy required to remove the atom from the surface but does not consider the kinetic energy of the sputtered atoms. Petrov et al[3], using the Yamamura et al[4] expression for the sputtering yield Y(E), showed that the maximum value of the erosion efficiency, η^{er}_{max}, exhibit periodic fluctuations as a function of the atomic number of the target Z_2 in the interval 0.4-4%.Here we extend this approach to calculate the sputtering energy efficiency $\eta^{sp} = (U+E_{av})Y(E)/E$, where E_{av} is the average kinetic energy of the sputtered atoms, estimated using the Thompson formula. Values of η^{sp}_{max} are approximately factor of x4 higher in the range of 2-16%. Ar delivers close to optimal total energy efficiency for targets with atomic number $Z_2 < 50$, while for heavier targets Kr yields higher total efficiencies from approximately 20% to 80%, and Xe from 20% - 110%. The sputtering energy efficiency η^{sp} exhibits maximum values at ion energies approximately factor of 2 higher than the values for η^{er}_{max}. The ion energy interval within which $\eta^{sp} > 0.8\ \eta^{sp}_{max}$ for most targets is 100-1500 eV.

1. M. Hans, J.M. Schneider, A. Matthews, C. Mitterer, Surf. Coat. Technol. **494**(2024)131486
2. G. Carter, M.J. Nobes and D.G. Armour, Vacuum **32**(1982)509
3. I. Petrov, V. Orlinov, S. Grudeva, Bulg. J. Phys. **18**(1991)215
4. Y. Yamamura, N. Matsunami, N. Itoh, Rad. Eff. **71**(1983)65

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

[2] **ASED Rising Star**

Electronic Materials and Photonics
Room 207 A W - Session EM2+CA+CPS+MS+SE+TF-WeM

Processing Ultra-Wide Band Gap Ga_2O_3

Moderator: Daniel Pennachio, Naval Research Laboratory

11:00am **EM2+CA+CPS+MS+SE+TF-WeM-13 Ga_2O_3 Polymorphs: Epitaxial Film Growth, Characterization and Contacts**, ***Lisa Porter***, *Jingyu Tang, Kunyao Jiang, Robert Davis, Posen Tseng, Rachel Kurchin,* Carnegie Mellon University*; Luke Lyle,* Penn State Applied Research Labs*; Carlo Schettini Mejia,* Carnegie Mellon University **INVITED**

The last decade has shown a dramatic increase in research on gallium oxide (Ga_2O_3) as an ultra-wide bandgap semiconductor for electronics that can operate in extreme conditions, such as high power, high temperature and radiation exposure. This presentation will focus on unique and intriguing characteristics associated with two processes that are necessary to produce Ga_2O_3-based devices: the growth of epitaxial films and the formation of ohmic and Schottky contacts. Whereas β-Ga_2O_3 is the thermodynamically stable phase, the other, metastable, phases of Ga_2O_3 can be produced as epitaxial films in either mixed-phase or pure-phase form. Our results, along with those in the literature, indicate that the phase content and other film properties strongly depend on the growth method (e.g., MOCVD, HVPE, mist CVD, etc.) and other conditions during film growth, such as precursor chemistry, flow rates, temperature, and substrate material / orientation. Our group has also conducted comprehensive studies of ohmic and Schottky contacts to β-Ga_2O_3. For reasons that are not well understood, only a few metals have been demonstrated as practical ohmic contacts to Ga_2O_3. Whereas Ti/Au contacts annealed at 400–500 °C are widely used, Cr/Au contacts annealed in a comparable temperature range also form ohmic contacts to Ga_2O_3. Controlled studies of several different elemental-metal Schottky contacts show that their electrical behavior highly depends on the particular Ga_2O_3 surface on which they're deposited; observed behavior ranges from Fermi-level pinning on the (-201) surface to near-ideal Schottky-Mott behavior on the (100) surface. Examples of the phenomena outlined above will be summarized and presented using results from high-resolution transmission electron microscopy, x-ray diffraction, and electrical measurements.

11:30am **EM2+CA+CPS+MS+SE+TF-WeM-15 Compensating Interfacial Parasitic Si Channels in β-Ga2O3 Thin Films Via Fe δ-doping**, ***Prescott Evans***, *Brenton Noesges, Jian Li, Mark Gordon, Daram Ramdin, Shin Mou, Adam Neal, Thaddeus Asel,* Air Force Research Laboratory, USA

β-Ga_2O_3 is a promising material for high power applications given an ultra-wide bandgap and predicted high break down field. One challenge with β-Ga_2O_3 for lateral device architectures is the presence of undesired Si between epitaxial thin film and substrate which creates a parasitic conduction channel. This channel limits performance and can prevent device modulation. Attempts to remove this interfacial layer using etch methods have proven mostly successful. However, in plasma-assisted oxide molecular beam epitaxy (PAMBE), conventional removal efforts appear unsuccessful. Our results show interfacial Si can reaccumulate at clean β-Ga_2O_3 surfaces from various Si sources inside the MBE tool such as the Si doping effusion cell. Hence, careful growth steps must be considered to avoid Si reaccumulating onto clean β-Ga_2O_3 surfaces in PAMBE. This work presents an alternative to mitigate the influence of this Si parasitic conduction channel via Fe delta doping at the interface. We demonstrate how a thin Fe layer at the interface can compensate interfacial Si and create an interface without excess free charge. The growth methodology presented involves multiple steps to avoid Fe diffusion from the interface. We first deposit the Fe followed by a low temperature (LT) undoped buffer before depositing an Si doped channel layer at higher deposition temperatures. The LT buffer helps minimize Fe surface riding and diffusion while the increased substrate temperature during the Si doped channel improves surface roughness. Secondary ion mass spectrometry (SIMS) results show Fe only resides at the interface between substrate and LT buffer layer with Fe concentration in the LT buffer and Si doped channel below the noise floor of the instrument. Furthermore, SIMS shows a smooth transition in Si concentration from the LT buffer into the intentionally Si-doped channel region avoiding any spikes between the two layers, indicating high degree of controlled doping localization. Initial capacitance-voltage (C-V) measurements on samples with the Fe compensation show no spike in carrier concentration near the substrate interface indicating Fe is fully compensating interfacial Si. These results demonstrate a potential method to mitigate parasitic Si conduction channels in β-Ga_2O_3. However, time-dependent C-V results show there is some capacitance transients when the sample is fully depleted. While Fe seems initially promising other compensating acceptors such as N or Mg need to be explored given this observation of capacitance transients in Fe-doped structures. Overall mitigating this parasitic interface will help improve yield and performance uniformity in fabricated devices.

11:45am **EM2+CA+CPS+MS+SE+TF-WeM-16 Investigating Metal Gate-Driven Interfacial Reactions in ALD-Grown Al_2O_3 on β-Ga_2O_3**, ***Joy Roy***, *Adam A. Gruszecki,* The University of Texas at Dallas*; Khushabu S. Agarwal, Paolo La Torraca, Karim Cherkaoui, Paul K. Hurley,* Tyndall National Institute, University College Cork, Ireland*; Chadwin D. Young, Robert M. Wallace,* University of Texas at Dallas

β-Ga_2O_3 is a leading candidate semiconductor for next generation power electronics with the potential to outperform GaN and SiC owing to its high breakdown strength paired with low power losses.[1] Integrating a robust gate dielectric and stable oxide interface is critical in leveraging these properties of β-Ga_2O_3.[2] However, this cannot be achieved without also considering the gate electrodes' reactivity and their influence on oxide properties. This work explores interfacial reactions—particularly those associated with oxygen scavenging—and the resulting variations in gate oxide performance induced by Ni and Ti gate metals in Al_2O_3 on bulk (001) β-Ga_2O_3 substrates.

Interface reactions were analyzed via *in situ* X-ray photoelectron spectroscopy (XPS) in an ultrahigh vacuum (UHV) cluster system. β-Ga_2O_3 samples were scanned as-loaded, after atomic layer deposition (ALD) of ~2 nm Al_2O_3, and a third time following UHV electron beam deposition of Ni or Ti (~1 nm) to assess changes in interface chemistries. Additional chemical states in Ga_2O_3 were below the XPS detection limit after oxide and metal deposition. However, an AlO_x (sub stoichiometric) state appeared in Al core levels (2*p* or 2*s*) after introducing Ti. This, along with a TiO_x state in Ti 2*p*, may imply oxygen scavenging from Al_2O_3. While both metals reacted with surface organic residues from metal-organic precursors, Ti exhibits more carbide formation at the gate/dielectric interface. Additionally, MOSCAPs were fabricated with ~12 nm Al_2O_3 and 10/100 nm of either Ni/Au or Ti/Au as the gate metal for I-V and C-V characterization. Ni/Au devices showed lower frequency dispersion and over two orders of magnitude lower gate leakage in accumulation than Ti/Au samples, consistent with the XPS findings. Dielectric breakdown strength will be further studied to explore electrical stability of the oxides.

In conclusion, a fundamental understanding of gate metals' influence on interface properties is essential for precisely predicting device behavior in power electronics.

This work was supported by the National Science Foundation (Grant ECCS 2154535) at the University of Texas at Dallas and by Research Ireland (Grant 12/US/3755) at Tyndall National Institute through the US-Ireland R&D Partnership. (Corresponding author: Robert M. Wallace.)

[1] S. J. Pearton, F. Ren, M. Tadjer, and J. Kim. J. Appl. Phys. **124**, 220901 (2018).

[2] C. V. Prasad, and Y.S. Rim, Mater. Today Phys. **27**, 100777 (2022).

Advanced Surface Engineering
Room 209 F W - Session SE-WeM

Advanced Surface Treatments for Enhanced Material Performance

Moderator: Diana Berman, University of North Texas

8:00am **SE-WeM-1 Tools for High-Throughput Autonomous Materials Discovery and Development for the Surface Engineer**, ***Christopher Muratore***, University of Dayton **INVITED**

The talk highlights automated experimental tools enabling synthesis and characterization of hundreds of samples per day. This approach, where experimentation is much faster than simulation has the potential to flip the traditional 'order of operations' for materials discovery where experiment feeds model during initial iterationsOne high-throughput format relies on scanning lasers with broad ranges of power, scan rates, and focal positions to induce physical and chemical transformations within materials. Laser heating parameters may be set to approximate quasi-equilibrium heating as in a furnace, or induce extreme heating and cooling rates, thereby broadening the range of accessible compositions and crystal structures dictated by kinetics of both chemical reactions and crystallization. Deposition tools, such as our magnetron sputtering system outfitted with

36 different source materials may also be used to create a broad range of compositions on the sample surface. Once a combinatorial sample with a desired range compositions and laser illumination conditions is processed, it can be manually or autonomously subjected to the combination of high-throughput characterization tools required for evaluation of the properties specified by the user. Autonomous systems enable users to specify a desired property and the system iterates processing and characterization data to 'make decisions' about optimization of conditions to realize the user-specified input.For example, an automated Raman spectroscopy system enables rapid collection of key data points (grain size, defect density, thickness, etc.) for technologically important optical, electronic, and energy materials.Some specific case studies include fundamental kinetics studies showing migration-limited crystallization kinetics amorphous materials can be directly observed.Pre-cursor materials for downstream processing can be converted directly into reaction intermediates with the appropriate non-equilibrium laser energy input to reduce process activation energy and process temperature required for high-quality materials. For photocatalysis materials rapid, non-equilibrium process conditions were identified demonstrating optimized performance with mixtures of phases.

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8:30am **SE-WeM-3 Hydrophobic and Hydrophilic Metallic Coatings: Their Sputter Depositions and Applications***, **Jinn P. Chu,*** National Taiwan University of Science and Technology, Taiwan

The talk will cover two types of metallic coatings with extreme properties: hydrophobicity and hydrophilicity.

First, an introduction to a low-friction hydrophobic metallic glass (MG) coating and its applications will be provided. This amorphous multicomponent coating, fabricated via sputter deposition, exhibits typical glass characteristics, such as a glass transition temperature upon heating. The MG coating has been successfully applied in various fields, including medical tools. This non-stick MG coating is intended to replace easily peeled-off teflon coatings, such as polytetrafluoroethylene (PTFE).

On the other hand, for the superhydrophilic coating, a 316 stainless steel layer is sputtered onto various substrates, resulting in a water contact angle of approximately 10 degrees on the coated surface. This coating also demonstrates antifouling and underwater superoleophobic properties, making it advantageous for use in separation membranes for oil/water emulsions. Moreover, it has proven highly effective in enhancing electrochemical responses in electrodes used for electrochemical sensors and supercapacitors.

8:45am **SE-WeM-4 Surface Engineering of Organic Nm-Thick PEDOT:PSS Films for Enhanced Electrical Conductivity***, Aaron DiFilippo,* Virginia Tech*; Amrita Chakraborty,* Virginia Tech, United States Minor Outlying Islands (the)*; **Marius Orlowski,*** Virginia Tech

We report on enhancing the electrical conductivity of Poly(3,4-ethylenedioxythiophene) Polystyrene Sulfonate (PEDOT:PSS) using various surface engineering methods, including acid treatment, topical doping with Cu and Ag nanoparticles, multilayer PEDOT:PSS deposition, and graphene incorporation. Our investigations reveal that optimizing multilayer deposition combined with nitric acid surface treatment yields superior results compared to alternative methods involving metal nanoparticles and graphene. This approach not only significantly enhances conductivity but also offers improved stability, reduced errors, and cost-effectiveness. Key optimization parameters, such as spinning speed, etchant concentration, and etching time, were identified as critical to achieving these outcomes. From all acids tested, nitric acid-treated multilayer PEDOT:PSS demonstrated a remarkable reduction in sheet resistance, from 1 MΩ/sq to 7 Ω/sq, corresponding to an increase in electrical conductivity from 0.18 S/cm to 15,699 S/cm—an improvement of over 10^5 times. Topical doping with Cu and Ag nanoparticles (30-90 nm) also improved conductivity, though less effectively than nitric acid treatment. Notably, Cu nanoparticles were as effective as Ag in topical doping, unlike bulk doping, where Cu oxidizes in aqueous solutions. This makes topical doping a versatile and cost-effective alternative, particularly for applications requiring surface metal nanoparticles, such as conductive dendrites for resistive RAM.

While each method individually enhanced conductivity, combining them did not yield significant additional improvements. The optimized nitric acid treatment, involving nine PEDOT:PSS layers, achieved the highest conductivity enhancement. However, acid-treated PEDOT:PSS exhibits a modest, self-limiting degradation in conductivity over time, stabilizing after a few days. This behavior suggests that while the initial enhancement is substantial, long-term stability requires further investigation.The study underscores the importance of optimization parameters and highlights the potential of these methods for advancing PEDOT:PSS-based technologies in flexible electronics, energy devices, and beyond. The surfaces have been characterized with Atomic Force Microsopy (AFM)X-ray diffraction (XRD), and optical microscopy in terms of surface roughness and surface composition. We also address film aging to mitigate reliability issues induced by ambient conditions.

9:00am **SE-WeM-5 Atomic Oxygen-Resistant Metal Oxide Coatings for Space Operations in Low-Earth Orbit***, **Joslin S. Prasanna**[1,2]**, Javier Meza-Arroyo, Minglei Sun, Chase Hazboun,* Department of Materials Science and Engineering, University of Texas at Dallas*; Fernando Quintero-Borbon,* Centro de Investigación en Materiales Avanzados S.C. (CIMAV), Unidad Monterrey, Mexico*; William Vandenberghe, Julia Hsu, Robert M. Wallace, Rafik Addou,* Department of Materials Science and Engineering, University of Texas at Dallas

Space operations in low-earth orbit (LEO) are hindered by the effect of atomic oxygen (AO) on spacecraft [1]. Coatings on spacecraft suffer loss of material due to erosion from attack by AO, thereby increasing drag and causing vehicles to drift from their operational orbit. Consequently, frequent thrust maneuvers are required to correct this position, consuming fuel, and ultimately limiting the duration of service. This raises the need to develop low-drag coatings for spacecraft that are resistant to AO and extend the life of space missions. Atomic-layer deposition (ALD) is an effective technique to deposit extremely conformal coatings of high quality and free from pinholes [2]. It also gives exceptional control over thickness down to the angstrom scale. Solution synthesis performed by the sol-gel method can scale the deposition process to deposit thicker films over larger areas.

In this work, we investigated the use of metal oxides (Al_2O_3 and TiO_2) deposited by ALD and sol-gel techniques as AO-resistant, low-drag coatings to protect conventionally used materials like Kapton and other polymers. SiO_2 and Kapton films were also studied alongside the metal oxide films for reference. Two different ALD recipes were tested for growing TiO_2, and one recipe for Al_2O_3. The films were characterized, both as deposited and after O_2 plasma exposure, by X-ray photoelectron spectroscopy (XPS) for chemical analysis, ellipsometry for thickness measurement, and atomic force microscopy (AFM) for surface roughness measurement. This investigation is further assisted by theoretical modeling of drag using experimentally determined surface structures. The drag simulations will be used to identify the key parameters influencing drag performance. Our study showed that Al_2O_3 deposited by ALD and sol-gel techniques displayed excellent resistance to O_2 plasma. The TiO_2 films showed more degradation but still far outperformed the Kapton and SiO_2 reference films, proving to be promising materials for AO-resistant coatings in LEO.

This work is supported by DARPA Materials Investigation for Novel Operations in Space (MINOS).

[1] S.W. Samwel et al., Space Res. J., 7(1), 1-13, (2014)

[2] T.K. Minton et al., Appl. Mater. Interfaces, 2(9), 2515-2520, (2010)

9:15am **SE-WeM-6 Reducing Tribological Run-in Through Morphology Control: A Dual-Layer Approach for Improved Environmental Insensitivity and Tribological Performance***, **Steven Larson,*** *Alex Mings, Tomas Babuska, Ping Lu, Jon Vogel, Michael Dugger, John Curry,* Sandia National Laboratories

Molybdenum disulfide (MoS_2)-based composite coatings are widely utilized in the aerospace and defense industries to reduce friction, prevent galling, and enhance wear resistance. These coatings are often grown as composites with various metals (Ni, Ti, Al, Pb, Au, WSe) and nonmetals (Sb_2O_3, PbO, C). A commonly used composition incorporates antimony oxide (Sb_2O_3) and gold (Au) into the MoS_2 matrix to promote increased density and improve wear life by amorphization. However, these additions significantly compromise the aging resistance of MoS_2, resulting in increased friction during both run-in and dwell-time phases.

In this presentation, we demonstrate a dual-layer MoS_2 sputtered coating designed to mitigate the effects of oxidation during long-term storage and short-term exposure to highly oxidizing environments, effectively transforming the traditionally water-sensitive MoS_2 into an environmentally insensitive coating. In addition to enhancing oxidation resistance, these structures facilitate a reduction in run-in friction, reducing initial cycle friction to near run-in levels (zero run-in coatings). We correlate deposition

[1] **ASED Rising Star**
[2] **JVST Highlighted Talk**

parameters with plasma probe measurements (retarding field energy analyzer), thin film material properties (density, crystallinity, hardness, modulus, and stoichiometry), and tribological performance (friction, run-in behavior, and wear rate) of the films. The resulting thin film stacks reduce initial friction after aging by nearly 300%, exhibiting negligible transient friction behavior (run-in) while maintain ultra-low wear rates.

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9:30am **SE-WeM-7 Optimizing Low-temperature MAX-phase Ti_2AlN Synthesis in Reactive TiAlN Multi-layer Thin Films**, ***Moses Nnaji***[1], *David Tavakoli,* Georgia Institute of Technology, USA*; Dale Hitchcock,* Savannah River National Laboratory*; Eric Vogel,* Georgia Institute of Technology, USA

$M_{n+1}AX_n$ phases are a class of layered nanolaminates where M represents a transition metal, A represents a group 12-16 element, and X represents carbon or nitrogen. These $M_{n+1}AX_n$ phases, or "MAX-phases", often boast a novel combination of metal and ceramic properties, including thermal and electrical conductivity, machinability, thermal shock resistance, and high-temperature integrity. Consequently, MAX-phase thin films have attracted interest for use in high-temperature protective coatings, Ohmic contacts, and low friction systems. However, the high temperatures (>700 °C) needed to form their complex crystal structures serve as a persistent bottleneck for meaningful adoption of MAX-phase coatings on sensitive substrates. Reducing the substrate temperature needed to form MAX-phase films is necessary to improve their viability in select applications, and thus improve the performance of said applications.

Magnetron sputtering is a common technique for MAX-phase thin film synthesis, and MAX-phase synthesis via low-temperature sputtering of reactive multi-layers and subsequent high-temperature annealing has also been reported. In this context, reactive multi-layers are thin film structures which can exploit exothermic reactions as a form of stored chemical energy. These reactions are not spontaneous at low substrate temperatures, but become favorable at higher temperatures and provide additional energy to the film. This phenomenon effectively results in phase transitions at decreased substrate temperatures. Thus, tailoring the thin film deposition process to yield multi-layers with high reactivity may help in forming MAX-phases at desirably low temperatures.

MAX-phase synthesis via reactive multi-layers is established, but work explicitly addressing the impact of the reactive multi-layer mechanism and phase composition on MAX-phase formation temperature is limited. Thus, comparing the behavior of composite multi-layers with different reactivities (e.g., containing constituent materials with different free energies and enthalpy) can provide insight towards minimizing the annealing temperature needed for MAX-phase synthesis. In this work, various techniques, including *in-situ* X-ray diffraction and differential scanning calorimetry, will be used to extensively characterize the evolution of MAX-phase Ti_2AlN as a function of annealing temperature in sputtered composite Ti-Al-N films. *In-situ* analysis of Ti/AlN, TiN/TiAl, and single-layer TiAlN thin film morphologies will aid optimization of processes that yield Ti_2AlN films at especially low temperatures.

9:45am **SE-WeM-8 Design of 2D Material-Based Coatings for Superlubricity in Sliding and Rolling Contacts**, ***Diana Berman***, *Ali Macknojia, Aditya Ayyagari,* University of North Texas

Friction and wear-related failures remain the greatest problems in today's moving mechanical components, from microelectromechanical devices to automotive assemblies and to biological systems. The critical need to reduce and eliminate the tribological failures constitutes the necessity for continuous search of novel materials and lubrication solutions. In this presentation, we demonstrate an experimental pathway to yield superlubricity in rolling-sliding contact conditions using MXene-based solid-lubricant materials. The material's compression and inter-layer shearing result in material reconstruction to pose superlubricity. High-resolution transmission electron microscopy analysis, complemented by multi-scan Raman spectroscopy showed the formation of a robust amorphous tribolayer. This demonstration is expected to not only advance the applied aspects in the development of oil-free solid lubricants but also push the boundaries of fundamental understanding of materials' structure-property relations across physical states.

11:00am **SE-WeM-13 Physics of Sample Charging During X-Ray Photoelectron Spectroscopy: Insights from Experiments with Thin Film Insulators**, ***Grzegorz (Greg) Greczynski***, Linköping University, Sweden **INVITED**

Sample charging during X-ray photoelectron spectroscopy (XPS) measurements of poorly conducting samples is a widely recognized concern that seriously complicates analysis of chemical bonding. The high complexity owing to many instrument- and sample-determined variables involved in the process is likely responsible for the fact that no comprehensive theory of charging exists. The present study aims to describe the development of charging for the case of thin insulating films supported on conducting substrates. Such systems are particularly well suited for studies of charging phenomena as they provide unique opportunity to separate effects that operate on different length scales and allow to investigate the role of charge supplied from the bottom contact. Two inherently insulating oxides, SiO_2 and WO_3, with the thickness varying by more than three orders of magnitude (from 1 to 5000 nm) are chosen to serve as model systems for insulators with respectively low and high X-ray-induced conductivity. The key role of low-energy secondary electrons (SE), X-ray penetration depth, sample work function, and the insulator SE yield in the development of surface charging is demonstrated. Based on these findings, a conceptual model is presented to serve as a starting point for the interpretation and discussion of charging phenomena in specific cases. Although the study is based on thin films the conclusions give insights into critical factors that govern charging phenomena in any other types of insulating samples.

11:30am **SE-WeM-15 Femtosecond Laser Ablation (fs-LA) XPS Depth Profiling for Surface Engineering**, ***Mark Baker***, *Charlie Chandler,* University of Surrey, U.K.*; Simon Bacon, Dhilan Devadasan,* Thermo Fisher Scientific, UK*; Oliver Parlour, Steve Hinder,* University of Surrey, U.K.*; Tim Nunney, Richard White,* Thermo Fisher Scientific, UK

For corrosion and wear resistance applications, XPS depth profiling is the most widely used analytical technique for chemical analysis of thin films, coatings and other surface treatments. The technique also provides important chemical information on corrosion and wear products, important in understanding degradation mechanisms. Traditional XPS sputter depth profiling has its advantages, such as good depth resolution, but leads to incorrect chemical compositions and chemical state information being recorded in the profile for many materials, due to ion beam induced damage. Sputtering is also a relatively slow process and the profiling depth is limited to approximately 5 µm for practical purposes. A new approach has recently been developed in which XPS depth profiles are generated through femtosecond laser ablation (fs-LA) rather than sputtering. This new methodology avoids chemical damage and has the ability to profile to much greater depths (many 10s microns) due to the effective instantaneous ablation process and the ability to easily vary the amount of material removed per pulse through changing the laser energy [1]. Using a 1030 nm wavelength, 160 fs pulsed laser, fs-LA XPS depth profiles will be shown for: (i) single and multi-layer thin films, coatings and other surface engineering processes employed to enhance corrosion and wear resistance; (ii) oxidised/corroded surfaces, demonstrating the capabilities of this new technique for surface engineering applications.

[1] M.A.Baker et al, *Applied Surface Science* **654** (2024) 159405

11:45am **SE-WeM-16 In Situ SEM Study of Graphene Rheotaxy: Growth on Molten Metals**, *Kristýna Bukvišová,* CEITEC; Thermo Fisher Scientific, Czechia*; Radek Kalousek,* Brno University of Technology, Czechia*; Jakub Zlámal,* CEITEC; BUT, Czechia*; Marek Patočka,* BUT, Czechia*; **Suneel Kodambaka**,* Virginia Tech*; Jakub Planer,* CEITEC, Czechia*; Vojtěch Mahel,* Thermo Fisher Scientific; BUT, Czechia*; Daniel Citterberg,* CEITEC, Czechia*; Libor Novák,* Thermo Fisher Scientific, Czechia*; Tomáš Šikola, Miroslav Kolíbal,* CEITEC; BUT, Czechia

Rheotaxy -- growth of crystalline layers on liquid substrates -- has been used to grow spatially-periodic self-assembled domains of graphene on molten metals such as Cu [1]. While earlier studies of graphene rheotaxy have identified the optimal growth parameters required for the growth of highly ordered domains, the mechanisms leading to self-assembly are not well understood [2,3]. Here, we present *in situ* scanning electron microscopy (SEM) studies of graphene growth via chemical vapor deposition of ethylene on molten Cu and Au surfaces at temperatures *T* between 1073 K and 1390 K.

The graphene layers are grown on solid and molten Cu ($T_{m,Cu}$ = 1357 K) in an ultrahigh vaccum (UHV) SEM and on solid and molten Au ($T_{m,Au}$ = 1336 K) in an environmental SEM equipped with a microReactor [4]. We observe

[1] **ASED Rising Star**

in situ the nucleation and growth of graphene domains, changes in their shapes and sizes as a function of deposition time, ethylene pressure, and the metal composition (Au or Cu), its state (solid or liquid), and temperature. From *ex situ* Raman spectroscopy data, we confirm that the as-deposited layers are graphene. Using *in situ* high-temperature atomic force microscopy (AFM) operated at ~1300 K, we measure the surface curvature of graphene. *In situ* SEM images acquired during the deposition of graphene reveal that graphene domains oscillate and self-assemble. We follow the dynamics of graphene domains on molten metal surfaces, measure amplitudes of domain fluctuations, and quantitatively determine the rate-limiting mechanisms controlling the graphene growth on solid and liquid Au and Cu surfaces. From the data, in combination with density functional theory (DFT) calculations and continuum modeling, we show that the graphene domain oscillations lead to self-assembly and are due to Casimir-like effect of surface undulations of the liquid metal [5].

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12:00pm **SE-WeM-17 Optical Metasurfaces in Iridium for High-Temperature Applications**, ***Zachary Kranefeld***[1], *T. Pan Menasuta, Kareena Guness, Kevin Grossklaus, Thomas Vandervelde,* Tufts University

Optical metasurfaces are gaining attention as a new technology because they can perform the tasks of several traditional optical elements simultaneously, while at a fraction of the size and weight. Their performance is dictated by the sub-wavelength structured surface, which also makes them extremely sensitive to morphology changes after fabrication. Oxidation and plastic deformation (edge rounding) are two dominant types of surface changes that occur in high-temperature environments, which limits optical metasurfaces from being a useful technology for those applications. We have pioneered a method of fabricating an optical metasurface into iridium films. Iridium is a refractory metal which is stable in these types of environments. The lack of reactivity, extremely high melting temperature, and high hardness are what makes iridium the best choice for the application. However, these features also present major challenges for lithographic pattern transfer at the nano-scale. The optical metasurface fabricated in our work was a selective emitter in the MWIR for a thermophotovoltaic system. The design process and all of the fabrication techniques we attempted are described, as well as the characterization of the final device.

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Advanced Surface Engineering
Room Ballroom BC - Session SE-ThP

Advanced Surface Engineering Poster Session

SE-ThP-1 Development of Multilayer Nano Nitride Layer for Corrosion and Wear Resistance by Using Magnetron Sputtering Technique, ***Aakanksha Jain***[1], indian institute of technology Roorkee, India; *Rahul S. Mulik, Ramesh Chandra,* INDIAN INSTITUTE OF TECHNOLOGY ROORKEE, India

This Study Investigates the Development of Multilayer Nano Nitride Coatings for Enhanced Corrosion and Wear Resistance, Fabricated Using the Magnetron Sputtering Technique. The Multilayer Coatings, Consisting of Alternating Thin Nitride Layers with Tailored Stoichiometries and Thicknesses, Are Designed to Improve Mechanical Properties and Protect Substrates from Aggressive Environments. The Corrosion Performance of the Coatings Was Assessed Using Electrochemical Impedance Spectroscopy (Eis), a Technique That Provides Valuable Insight Into the Electrochemical Behavior and Protective Efficiency of the Coatings in Corrosive Media. The Eis Results Demonstrated a Marked Improvement in the Corrosion Resistance of the Multilayer Coatings Compared to Uncoated Substrates and Single-Layer Coatings, Indicating Their Superior Ability to Act as a Barrier Against Corrosive Agents.

Nanoindentation Was Employed to Evaluate the Mechanical Properties, Particularly the Hardness of the Coatings. This Technique Allowed for Precise Hardness Measurements at the Nanoscale, Revealing a Significant Increase in Hardness for the Multilayer Coatings Compared to Both the Substrate and Single-Layer Nitride Coatings. The Improved Hardness Is Attributed to the Unique Microstructure and the Stress Distribution Across the Multilayer Design, Which Enhances Wear Resistance and Mechanical Durability.

the Coatings' Microstructure, Phase Composition, and Adhesion Strength Were Further Characterized by X-Ray Diffraction (Xrd), Scanning Electron Microscopy (Sem), and Scratch Testing, Confirming the High Quality of the Multilayer Coatings and Their Strong Interlayer Bonding. Overall, the Multilayer Nitride Coatings Exhibited Enhanced Mechanical and Electrochemical Properties, Providing Excellent Corrosion and Wear Resistance. These Results Suggest That Magnetron Sputtering, Combined with Eis and Nanoindentation, Is an Effective Approach for Developing Advanced Nitride Coatings Suitable for Applications in Harsh Industrial Environments Requiring High Durability and Long-Term Performance. This Study Will Help in Marine Applications for Future Purposes and Be Beneficial.

Keywords: Magnetron Sputtering, Nitride Coatings, Hardness, Corrosion, Marine Application.

SE-ThP-2 Geometry Matters in Magnetron Sputtering: From Source Placement to Film Quality, ***Esteban Broitman***, *Rickmer Kose, Sven Kelling,* SENTYS Inc.

Magnetron sputtering remains a premier thin-film deposition technique, with its performance critically dependent on the spatial relationship between magnetron cathodes and the target–substrate assembly. In this review, we collate experimental findings from the literature into three principal geometric classifications: sputter-up versus sputter-down magnetron orientations, planar versus confocal magnetron arrangements, and on-axis versus off-axis substrate positioning.

For each geometric class, we assess how the arrangement influences plasma confinement, governs the angular dispersion of sputtered species, and ultimately controls key film attributes—including mass density, residual stress, microstructural development, and step-coverage uniformity. By establishing quantitative correlations between spatial parameters and these film properties, we formulate a design-oriented framework to aid researchers in choosing the optimal magnetron configuration for targeted material performance.

To demonstrate practical application, we highlight state-of-the-art deposition chambers and magnetron source designs that permit rapid, tool-free reconfiguration of source–substrate geometry. Such adaptable platforms enable real-time tuning of deposition conditions, granting precise control over thin-film growth in situ.

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