

Advanced Surface Engineering Room 303 - Session SE-WeM

Advanced Surface Engineering I

Moderators: *Diana Berman*, University of North Texas, *Ivan Petrov*, University of Illinois at Urbana-Champaign

8:00am SE-WeM-1 Tailoring the Performance of MXene-Reinforced PEEK Composite Coatings through Optimised Powder Mixing Strategies, Mikdat Gurtaran, A. Madhan Kumar, King Fahd University of Petroleum and Minerals, Saudi Arabia

Poly(ether ether ketone) (PEEK) is a high-performance thermoplastic extensively used in engineering applications, primarily biomedical, owing to its excellent mechanical properties, thermal stability, and chemical resistance. The incorporation of two-dimensional MXene nanosheets has emerged as an effective approach to further enhance the surface, mechanical and corrosion performance of PEEK composite coatings. Nevertheless, the effectiveness of MXene reinforcement is strongly dependent on achieving a uniform dispersion within the polymer matrix. This study systematically investigates the influence of three different mixing techniques on the microstructure and surface properties of MXene-reinforced PEEK composite coatings on carbon steel substrates.

PEEK and MXene nanoparticles were blended using three different approaches: (i) manual hand mixing, (ii) ball milling using polymer milling media with a ball-to-powder ratio of 1:11 at 100 rpm for 30 min with alternating 5 min milling and 5 min rest intervals to prevent excessive heating, and (iii) solvent-assisted mixing, in which the powders were dispersed in ethanol under magnetic stirring overnight followed by solvent removal at 100 °C. The prepared MXene/PEEK powders were subsequently deposited onto carbon steel substrates using an electrostatic powder spray gun and thermally cured at 375 °C for 30 min to produce dense composite coatings. The coatings were characterised using scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), and X-ray diffraction (XRD). Nanoindentation, progressive-load scratch testing, and electrochemical corrosion measurements were performed to evaluate the mechanical, tribological, and corrosion performance of the coatings.

The results demonstrate that the selected mixing strategy significantly influences MXene dispersion and the resulting microstructure, which directly affects the surface performance of the composites. Among the investigated approaches, the solvent-assisted mixing route produced the most homogeneous distribution of MXene throughout the PEEK matrix, resulting in superior hardness, improved scratch resistance, and enhanced corrosion resistance compared with the hand-mixed and ball-milled specimens. These improvements are attributed to the more effective deagglomeration of MXene nanosheets and stronger interfacial interactions between the reinforcement and the polymer matrix. This study highlights the critical role of powder processing in determining the performance of MXene/PEEK composite coatings and provides practical guidance for the fabrication of high-performance polymer nanocomposites for advanced engineering applications.

8:15am SE-WeM-2 Friction and Wear of Composite MXene/MoS₂ Coating Under Low-Viscosity Fuels, Ali Zayaan Macknoja¹, Diana Berman, Andrey Voevodin, Samir Aouadi, University of North Texas; *Stephen Berkebile*, Army Research Laboratory

Friction and wear-related failures remain major challenges in moving mechanical assemblies operating under various conditions. For example, the components of fuel systems made of AISI 52100 steel are susceptible to scuffing-induced wear when operated in a fuel environment. This study demonstrates the decreased friction and wear characteristics achieved by spray-coating 52100-grade steel surfaces with solution-processed multilayer Ti₃C₂Tx-MoS₂ blends. The study analyzed the performance of the coating under high contact stresses and sliding speeds in different fuels. Raman spectroscopy, scanning electron microscopy, and transmission electron microscopy results revealed the formation of a robust in situ 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 environments, inspiring further research and development in this field.

8:30am SE-WeM-3 Fabrication of CrMoNbWTiC_x High Entropy Alloy Sublattice Carbide Films by High Power Impulse Magnetron Sputtering System: Effect of Target Poisoning, Jyh-Wei Lee, Ming Chi University of Technology, Taiwan; *Chun-Hao Cheng*, *Yung-Chin Yang*, National Taipei University of Technology, Taiwan; *Bih-Show Lou*, Chang Gung University, Taiwan; *Chia-Lin Li*, Ming Chi University of Technology, Taiwan

High entropy alloys have been widely studied due to their unique properties and potential applications since they were reported by Prof. J.W. Yeh and Prof. B. Cantor, respectively, in 2004. The research on HEA thin films by magnetron sputtering also attracted lots of attention because of its flexibility to deposit on various substrates. Highpower impulse magnetron sputtering (HiPIMS) is characterized by its short pulses, high peak power density, and low duty cycle, enabling the generation of high-density plasma and a high degree of metal ionization. This enhances ion bombardment, thereby improving film densification and adhesion. In this study, CrMoNbWTiC_x/HEA sublattice carbide (HEASC) films were deposited by HiPIMS using a plasma emission monitoring feedback control system under different Ar and C₂H₂ mixed gas environments. Effects of target poisoning and carbon content on the phase structure, mechanical, and tribological properties of HEASC films were studied. Optical emission spectroscopy (OES) revealed that increasing C₂H₂ flow decreased Cr emission intensity, indicating a transition from metallic to poisoned mode, with clear hysteresis behavior. With increasing carbon content from 32.7 to 77.3 at.%, the HEASC films exhibited grain refinement and eventually transformed from FCC to an amorphous structure. The C40 film with 40.9 at.% C showed the highest hardness of 30.4 GPa and optimal H/E₁ and H³/E₁² values. For thin films with higher than 61.3 at.% carbon content exhibited reduced coefficient of friction values of ~0.23–0.32 and improved wear resistance due to the lubricating effect of amorphous carbon. We can conclude that an intermediate carbon content, from 36.8 to 45.1 at.% C, provides the best balance between mechanical and tribological performance of CrMoNbWTiC_x/HEASC films.

8:45am SE-WeM-4 Combinatorial Sputter Deposition of Low-Emissivity Metal Binaries for Extreme Environments, Ryan Spangler, Kyle Dorman, Finley Haines, Cedric Shaskey, Anthony McDonald, Brooklynn Nash, Toai Ton-That, Amun Jarzembski, David Adams, Sandia National Laboratory

Certain nanocrystalline metal alloys, such as the Pt_xAu_{1-x} solid solution, exhibit both low infrared emissivity and high thermomechanical stability. These systems may thus be useful as coatings to minimize radiative heat transfer in extreme environments; however, applications require the exploration of a large compositional and process space. Combinatorial thin-film synthesis, when paired with high-throughput characterization, can significantly accelerate materials exploration and process development. In this work, we use simulation (SIMTRA)-guided off-axis combinatorial sputter deposition to rapidly synthesize hundreds of compositions, thoroughly mapping out Pt_xAu_{1-x}, Ni_xAu_{1-x}, and other binary metal systems. We measure the physical properties—including thickness, density, and resistivity—of each composition using optical interferometry, X-ray reflectivity, and four-point probe methods. We then develop a mapping, two-temperature radiance method to rapidly measure the low infrared emissivity values of the combinatorial films with high spatial resolution. We explore the strong correlation between the infrared emissivity of these compositions and their resistivity, which may enable the use of one measurement to predict the other. By building out a multi-dimensional processing-structure-property library, favorable compositions and growth conditions may be selected that meet the optical, mechanical, and other demands for specific applications.

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

9:00am SE-WeM-5 Ground-Based VLEO Assessment and SiO₂ Protection of FFF-Printed Onyx and Onyx ESD Composites, Jihyun Kim, Korea Advanced Institute of Science and Technology (KAIST), Republic of Korea; *Jungryul Lee*, Korea Advanced Institute of Science and Technology (KAIST), Korea (Democratic People's Republic of)

Fused filament fabrication (FFF) enables rapid fabrication of thermoplastic-composite spacecraft components. However, their durability under VLEO environments remains a challenge. In this study, carbon-fiber-reinforced nylon composites (Onyx and conductive Onyx ESD) fabricated by FFF were evaluated for atomic-oxygen (AO) durability and SiO₂ protection. Coupons were tested on Top and Bed-contact surfaces under sequential thermal cycling, UV-C, vacuum, AO, and 20 keV electron exposure. The cumulative Kapton-equivalent AO fluence was 9.13×10^{21} atoms cm⁻², matched to a

one-year reference mission at 365.5 km; other factors used accelerated or facility-attainable conditions.

Stage-resolved SEM revealed that the largest morphology change occurred during accelerated AO exposure, producing preferential recession of the PA6-rich matrix, progressive short-carbon-fiber exposure, and heterogeneous surface texturing. These changes are attributed to AO-induced oxidative chain scission and volatile product removal, degrading the polymer-rich phase and exposing the erosion-resistant carbon reinforcement. EDS confirmed relative oxygen enrichment on all uncoated surfaces, with the mean O/C ratio increasing approximately twofold after exposure. Apparent erosion yields were $2.59\text{--}2.72 \times 10^{-24} \text{ cm}^3\text{atom}^{-1}$ for Onyx and $2.44\text{--}2.57 \times 10^{-24} \text{ cm}^3\text{atom}^{-1}$ for Onyx ESD. All values fell within the $1.41\text{--}2.94 \times 10^{-24} \text{ cm}^3\text{atom}^{-1}$ PA6/carbon sensitivity range estimated using MISSE-2 PA6 references and the NASA erosion-resistance mixture relation, supporting a physically consistent response.

Nominal 100–400 nm SiO₂ films were deposited by RF sputtering without intentional substrate heating. Increasing coating thickness produced a stronger SiO₂-associated near-surface contribution before exposure. After exposure, residual Si signals became reduced and spatially heterogeneous, while SEM showed preserved and locally eroded regions even at 400 nm. Nevertheless, whole-coupon apparent erosion yield decreased monotonically with thickness in all four material–surface series. At 400 nm, the response was reduced by 56.3–78.7% relative to uncoated surfaces, with a minimum of $0.519 \times 10^{-24} \text{ cm}^3\text{atom}^{-1}$ for Onyx ESD.

The measured degradation was incorporated into a provisional mission-specific wall-loss framework separating whole-coupon mass-derived response from local recession. The framework links end-of-life recession demand with structural and functional requirements, enabling coating-thickness selection after direct recession and retained-property validation. Thus, 400 nm represents the best-performing candidate within the tested range rather than an optimum or minimum required thickness.

9:15am SE-WeM-6 TiZrN-Based Coatings for Dry Machining Deposited by Dual-Beam EB-PVD, Andre Giordimaina, Kieran Axisa, Glenn Cassar, University of Malta

TiZrN-based hard coatings are attractive candidates for dry machining applications, but their deposition via electron-beam physical vapour deposition is typically limited by markedly different evaporation rates between Ti and Zr. In this study, TiZrN-based coatings were developed on AISI H13 (DIN 1.2344) steel substrates using a plasma-assisted dual-beam EB-PVD process. Such coatings were investigated for the long-term objective of enabling multifunctional Ti-Zr coating systems, such as TiZrCrN, but initial trials showed preferential Ti evaporation from the melt pool, generating a Ti-rich vapour flux and restricting Zr incorporation. To avoid Zr-depleted coatings, Ti-Zr targets with varied Ti:Zr ratios were investigated to compensate for the differential evaporation and to achieve compositionally controlled TiZrN-based coatings. The deposited coatings were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX) nanoindentation and pin-on-disk tribology, with emphasis on the correlation between composition, microstructure, mechanical properties, and wear behaviour relevant to dry-machining conditions. By treating source chemistry as an active process-control variable, the study aims at providing an EB route towards compositionally controlled TiZrN-based coatings for lubricant-free machining.

9:30am SE-WeM-7 Fundamentals of Thin Film Growth Dynamics: Advances in Hybridizing CFUBMS and HiPIMS, Esteban Broitman, Sentys, Sweden; Hailin Sun, Teer Coatings Ltd, UK

Control over the energy and flux of species arriving at a substrate is a fundamental requirement for tailoring thin film growth and macroscopic properties. Traditional methods, including Balanced Magnetron Sputtering (BMS) and standalone Unbalanced Magnetron Sputtering (UBMS), face critical plasma confinement limits that result in porous, columnar microstructures. Closed-Field Unbalanced Magnetron Sputtering (CFUBMS), originally invented and patented by D. G. Teer in 1991, solves this by linking neighboring unbalanced magnetrons with opposing polarities. This configuration creates a closed magnetic trap that delivers dense, high-flux, low-energy ion bombardment across the substrate. This intense atomic peening eliminates open voids and transitions film growth from porous columns into highly dense, equiaxed structures.

In the first part, this review presents direct comparative examples evaluating film growth under BMS, UBMS, and CFUBMS modes. Crucially, in the second part, this review demonstrates that the scientific community no

longer views CFUBMS and High-Power Impulse Magnetron Sputtering (HiPIMS) as mutually exclusive technologies. Instead, state-of-the-art industrial systems are increasingly hybridizing them. This approach integrates pulsed-DC CFUBMS architectures to preserve fast deposition rates and minimize substrate thermal loads, while simultaneously overlaying advanced HiPIMS pulses to achieve the ultra-high metal ionization required for stress-relaxed, column-free structures. Recent case studies across mechanical, tribological, electrical, and optical properties illustrate how transitioning to these modern, hybridized industrial configurations alters fundamental grain growth. By mapping these mechanisms, this presentation delivers an essential engineering framework for leveraging fundamental thin-film physics to optimize throughput and performance in commercial thin-film manufacturing.

9:45am SE-WeM-8 Guinier-Preston Zone Hardening and Thermal Stability of Metastable NaCl-Structure Refractory Nitrides, Ivan Petrov, University of Illinois; Vladyslav Rogoz, Sanjay Kumar, Linköping University, Sweden; Marcus Hans, Jochen M. Schneider, RWTH Aachen University, Germany; Lars Hultman, Linköping University, Sweden, Ming Chi University of Technology, Taiwan, Sweden; Davide G. Sangiovanni, Grzegorz Greczynski, Linköping University, Sweden

Recently, atomic-plane-thick W-based Guinier–Preston (GP) zones have been reported to form on (111) planes in metastable NaCl-structure (B1) TiAlWN leading to age hardening at temperatures relevant to high-speed cutting applications. Here we compare how the addition of transition metals (M) = Hf, Ta, and W modifies the high temperature behavior of TiAlMN thin films.

We find that the phase separation during annealing occurs via competing mechanisms: spinodal decomposition or nucleation-and-growth of second equilibrium phase, the latter including the special case of Guinier-Preston (GP) zone formation. After 2 h annealing at 950 °C Hf and Ta-doped systems exhibit spinodal decomposition into NaCl-structure (B1) c-(Ti,Hf)N and c-AlN enhancing hardening (ΔH) by 4.8%, and c-(Ti,Ta)N plus c-AlN phases with $\Delta H = 10.9\%$, respectively. W-doped systems, however, form coherent GP zones in addition to spinodal decomposition achieving maximum hardening ($\Delta H = 19.7\%$). DFT calculations confirm thermodynamic preference for GP zone formation in the W case by virtue of low interfacial energy and strain energy minimization to the matrix.

In addition, the generality of GP-zone formation is explored for a range of related quasi-binary and -ternary W-containing nitrides. Cubic-phase M_eWN and M_eAlWN films (M_e = V, Cr, Zr) are deposited by hybrid HiPIMS/DCMS co-sputtering. High-resolution TEM/STEM reveals GP zones even in AlN-free systems, V_{0.74}W_{0.26}N and Cr_{0.70}W_{0.30}N. Their volume density is, however, higher in AlN-containing counterparts: V_{0.45}Al_{0.28}W_{0.27}N and Cr_{0.39}Al_{0.31}W_{0.30}N. No GP zones are observed in Zr_{0.70}W_{0.30}N and Zr_{0.41}Al_{0.29}W_{0.30}N, both characterized by larger lattice parameter.

These results indicate that the tendency for spinodal decomposition in AlN-containing films promotes GP zone formation but is not a necessary condition. Minimization of the coherency strain between the (111) planes of the host matrix and GP zone together with reduction of the local compressive stress introduced by incorporation of WN into the host matrix are proposed as a driving force leading to W clustering. GP zones hinder dislocation movements under external load, which leads to strengthening effect provided their volume density is high. Importantly, GP zone hardening occurs at annealing temperatures higher than spinodal decomposition requires, which brings promise to extending cutting tool operational temperatures to 1000 °C and above. The magnitude of GP-zone age hardening can be readily controlled through the alloy composition, thus providing additional degrees of freedom in the design of novel materials compared to present practice.

Advanced Surface Engineering Room 303 - Session SE-WeA

Advanced Surface Engineering II

Moderators: Diana Berman, University of North Texas, Jyh-Wei Lee, Ming Chi University of Technology

4:15pm SE-WeA-9 ASSD Student Award Finalist Presentation: In-Situ Tribo-Sintering and Atomic Structure of 2D Silicate Interfacial Films derived from Sustainable Oleogels, Mohammad Eskandari¹, Diana Berman, Ali Zayaan Macknoja, University of North Texas

Designing stimuli-responsive interfacial materials capable of adapting to extreme thermomechanical stresses remains a profound challenge in surface science. Conventional liquid lubricants typically exhibit catastrophic boundary film failure at elevated temperatures. Here, we report a paradoxical friction-reversing phenomenon in sustainable bio-oleogels thickened with organically modified montmorillonite nanoclay: an autonomous transition to a macroscopic superlubricity regime (COF <0.01) initiated solely by high contact pressures (500 N) and thermal activation (150°C).

To unravel the structural mechanisms underlying the observed superlubricity phenomena, we first performed a post-tribology Raman spectroscopy analysis that confirmed the formation of a clay-based tribofilm. The 3D topographical maps acquired with ultra-high-resolution confocal laser scanning microscopy (~140 nm) confirmed the contiguous nature of it. Electron diffraction and lattice fringe imaging using high-resolution transmission electron microscopy (HR-XTEM) revealed the in-situ self-assembly of ordered layers, sintered at the plastically deformed metallic asperities. Through the combined multi-scale microscopy approach, this research provides direct evidence of a shear-induced sol-to-solid phase transition at the sliding interfaces, revealing a new class of intelligent, bio-derived, adaptive liquid interfaces for extreme environments.

4:30pm SE-WeA-10 Development and Characterization of BaTiO₃-GO reinforced PEEK Nanocomposites for High Temperature Functional Applications, Unnati Joshi, Anand Joshi, Sushila Vadu, Parul University, India

Polyether ether ketone (PEEK) has emerged as a high performance engineering thermoplastic due to its excellent mechanical strength, thermal stability, chemical resistance, and biocompatibility. However, its relatively low thermal conductivity and limited functional properties restrict its application in advanced electronic, aerospace, and tribological systems. In the present study, Barium Titanate (BaTiO₃) and Graphene Oxide (GO) reinforced PEEK nanocomposite was synthesized to investigate the synergistic influence of ceramic and carbon-based nanofillers on the thermal and microstructural characteristics of the polymer matrix.

The nanocomposite was prepared through Solution Processing and Homogenization of the nanomaterials, ensuring uniform dispersion of the reinforcing phases within the PEEK matrix.

Microstructural characterization was performed using scanning electron microscopy, which revealed a relatively homogeneous distribution of BaTiO₃ particles and GO powder with limited agglomeration. Energy-dispersive X-ray spectroscopy confirmed the presence and elemental distribution of Ba, Ti, C, and O within the composite structure, validating successful incorporation of the reinforcing phases. X-ray diffraction analysis identified the characteristic crystalline peaks of PEEK and BaTiO₃ while also indicating structural interactions between the fillers and matrix.

The thermal behaviour of the proposed nanocomposite was evaluated by STA from 25–1000 °C. The composite retained 98.6% and 95.4% mass at 300 and 500 °C, respectively, indicating excellent thermal stability. Major degradation occurred between 525–625 °C, with the maximum mass-loss rate at approximately 573 °C. The delayed degradation and substantial high-temperature residue are attributed to the synergistic thermal-barrier effects of GO and thermally stable BaTiO₃ reinforcements.

The combined thermal and structural analyses demonstrate that BaTiO₃-GO reinforced PEEK nanocomposites exhibit enhanced thermal performance and stable microstructural characteristics, making them promising candidates for multifunctional applications such as dielectric

components, electronic packaging, aerospace structures, insulated bearings, and high temperature engineering systems.

4:45pm SE-WeA-11 Mechanochemically-Driven Formation of Zero-Friction and Zero-Wear Lubricating Carbon Films, Diana Berman, University of North Texas

Mechanically-induced surface degradation creates a significant problem for currently-used mechanical assemblies and is the major cause for their loss of usefulness. Meanwhile, high-contact pressure and shear during relative movement of the sliding interfaces provide the unique capability for local heating and shear- and load-induced compression of the sliding surfaces. For a correct combination of materials in sliding contact, these conditions may induce tribochemical reactions that lead to the formation of a protective damage-suppressing tribofilm directly at the contact. In this presentation, we overview recent advances in establishing the fundamental understanding of materials interactions at sliding interfaces and use this knowledge as a guide to developing nanomaterials solutions that enhance reliability and efficiency of tribological systems. We demonstrate tribochemically-driven self-replenishment of materials inside the contact interfaces, thus enabling a zero-wear sliding regime.

Overall, the findings could open a new avenue for the development of new concepts and design strategies for next generation of tribologically efficient materials systems.

¹ ASSD Student Award Finalist

Advanced Surface Engineering Room 304 - Session SE-ThA

Advanced Surface Engineering III

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

2:15pm SE-ThA-1 In-Situ Raman Spectroscopy of Adaptive Tribological Coatings, **Andrey Voevodin**, University of North Texas **INVITED**

In-situ Raman spectroscopy is a powerful method to investigate adaptive tribological surfaces. The in-situ Raman spectroscopy is shown to capture tribologically induced changes of adaptive coatings inside the lubricated sliding contact in response to applied mechanical and thermal stresses. For solid lubricated adaptive contacts, duplex plasma-electrolytic oxidation (PEO) and chameleon coating produced on Al alloys were investigated. These were composed of a 80-160 nm thick hard load-supporting Al-Si-O coating and a 5-8 μm top layer of a chameleon coating made of graphite or BN, MoS_2 or WS_2 , and Sb_2O_3 . For liquid contacts, 52100 steel coupons were tested under lubricant starvation regimes using low viscosity hydrocarbons. The counterpart pins were made of 52100 steel and Si_3N_4 . The tests were performed at temperatures ranging from room (liquid lubrication) to 600 °C (solid lubrication). In-situ Raman spectroscopy revealed that the lubricating phases, i.e. MoS_2 , WS_2 , and graphite, were protected from oxidation by the porous PEO structure. It also revealed a gradual evolution of chameleon composition with diminishing orthorhombic Sb_2O_3 phase presence at the surface in favor of hexagonal lubricating phases of chameleon components. The low shear strength of MoS_2 , WS_2 , and graphite and the integration of the chameleon coating with the PEO sublayer were responsible for the ultra-low friction behavior. In all examples, the in-situ Raman spectroscopy was shown to reliably detect contact chemical change correlated with the observed friction behavior.

2:45pm SE-ThA-3 Automated Characterization of Combinatorial Thin Films and Their Stress, **David Adams**, **Finley Haines**, **Amun Jarzembksi**, Sandia National Laboratories, USA

Combinatorial sputter deposition techniques provide an ability to fabricate a large variety of thin films in a relatively short time thereby accelerating materials discovery. Indeed, past studies have reported methods that produce 10s or 100s of unique films in a single vacuum deposition. Despite these advances, new, high-throughput characterization techniques are needed to match the pace of combinatorial synthesis. In particular, automated techniques that accurately reveal the structure and the relevant properties of combinatorial films are of great interest.

Toward this end, we describe automated, ex-situ characterization of biaxial film stress wherein >100 individual, combinatorial thin films are probed in concert. Combinatorial, magnetron sputter-deposited Pt-Au and Pt-Ni films are interrogated using a k-Space Co. Thermal Scan instrument wherein samples are affixed inside a vacuum test cell. This instrument precisely moves a laser spot array to each specimen (before and after deposition) to determine wafer curvature changes. The application of Stoney's equation to determine the stress of combinatorial films from measured curvatures is discussed. Films are much thinner than the substrates (which satisfies one Stoney assumption), but a laterally graded thickness across each specimen moves away from at least one separate assumption used in the derivation of this often-used equation. Accounting for measured thickness gradients, we demonstrate accurate stress measurements over a range of temperatures up to +600C. Combinatorial Pt-Au and Pt-Ni films produced in single depositions exhibit a variety of stress responses that are ultimately correlated to measured film density and composition. Altogether, we determine their thermal expansion coefficients, pinpoint their onset of relaxation, and reveal changes occurring at high temperature tied to secondary phase formation. The gathered information augments an extensive library of process-structure-property relationships that underly their high-temperature performance.

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

3:00pm SE-ThA-4 Designing Defect Structure and Interfacial Strain in an Epitaxial VN Bilayer Film by Tailoring N Concentration, **Marcus Hans, **Damian Holzapfel**, RWTH Aachen University, Germany; **Zhuo Chen**, **Erich Schmid** Institute of Materials Science, Austria; **Soheil Karimi Aghda**, **Michal Fečík**, RWTH Aachen University, Germany; **Daniel Primetzhofer**, Uppsala University, Sweden; **Zaoli Zhang**, **Erich Schmid** Institute of Materials Science, Austria; **Jochen Schneider**, RWTH Aachen University, Germany**

A bilayer of $\text{V}_{0.49}\text{N}_{0.51}/\text{V}_{0.56}\text{N}_{0.44}$ has been grown epitaxially on $\text{MgO}(001)$ by reactive high power pulsed magnetron sputtering in an industrial-scale deposition system at a temperature of 400 °C and it is demonstrated that the defect structure and interfacial strain are governed by the N concentration. Based on the lattice mismatch between MgO and $\text{V}_{0.49}\text{N}_{0.51}$ with V vacancies, an interfacial strain of -2.3(1)% is expected. From ab initio calculations, X-ray diffraction and transmission electron microscopy data it is inferred, that the $\text{V}_{0.49}\text{N}_{0.51}$ layer exhibits V vacancies, N Frenkel pairs and a high dislocation density of $\sim 0.20 \text{ nm}^{-2}$, causing an interfacial strain of -1.4(5)% at the $\text{MgO}/\text{V}_{0.49}\text{N}_{0.51}$ interface. The phase formation of understoichiometric $\text{V}_{0.56}\text{N}_{0.44}$ is governed by N vacancy formation, while the dislocation density is reduced to $\sim 0.04 \text{ nm}^{-2}$ at the $\text{V}_{0.49}\text{N}_{0.51}/\text{V}_{0.56}\text{N}_{0.44}$ interface and to $< 0.01 \text{ nm}^{-2}$ within $\text{V}_{0.56}\text{N}_{0.44}$ at a distance of $\sim 35 \text{ nm}$ from the interface. Based on ab initio calculations, a strain of -1.7(6)% is predicted at the $\text{V}_{0.49}\text{N}_{0.51}/\text{V}_{0.56}\text{N}_{0.44}$ interface in very good agreement with the experimentally obtained value of -1.6(8)%. It is evident that control of the N concentration allows for the design of layered architectures with well-defined strained interfaces and tailored defect structures. Finally, the employment of only one transition metal is chemically simple and therefore, from a recycling point of view, also more sustainable than combining different transition metals as commonly done in layered architectures, including superlattices.

3:15pm SE-ThA-5 Corrosion Behaviour of Zr-Ti-N Thin Films, **Jyoti Menghani**, Sardar Vallabhbhai National Institute of Technology SURAT, India; **k Baba Pai**, ITM UNIVERSE, India; **Nitin Jalgaonkar**, Seco Tools ,India

Zr-Ti-N coatings with varying thicknesses (1.5, 2.0, 2.5, and 3.0 μm) were deposited on 316 stainless steel substrates by cathodic arc evaporation using separate zirconium and titanium targets in a reactive nitrogen atmosphere. The influence of coating thickness on the microstructure and corrosion behavior of the films was systematically investigated. X-ray diffraction (XRD) analysis revealed the formation of crystalline Zr-Ti-N phases along with the presence of substoichiometric Ti_2N .

Surface and cross-sectional morphologies of the coatings were examined using scanning electron microscopy (SEM), which confirmed dense columnar growth of the deposited films. Cathodic arc-induced droplets were observed on the coating surfaces, and their presence was found to influence surface morphology and localized corrosion characteristics. Variations in coating thickness significantly affected coating compactness, defect density, and structural integrity.

The corrosion behavior of the coatings was evaluated in 0.1 N HCl solution using potentiodynamic polarization and electrochemical impedance spectroscopy (EIS). The acidic HCl medium was selected to simulate aggressive industrial corrosive environments. Electrochemical studies demonstrated that the corrosion resistance of the coatings improved with increasing coating thickness. The corrosion current density (I_{corr}) decreased from $136 \mu\text{A}/\text{cm}^2$ for 1.5 μm Zr-Ti-N coating to $92.14 \mu\text{A}/\text{cm}^2$ for 3.0 μm coating, indicating enhanced barrier protection and reduced electrolyte penetration. EIS results further confirmed the improvement in charge transfer resistance and protective efficiency with increasing coating thickness.

The study demonstrates that coating thickness, microstructural evolution, and cathodic arc droplet formation play a significant role in determining the corrosion performance of cathodic arc deposited Zr-Ti-N coatings for protective applications in aggressive acidic environments.

3:30pm SE-ThA-6 High Throughput Thin Film Materials Synthesis via Laser Spike Annealing, **Drake Austin**, Air Force Research Laboratory (AFRL) **INVITED**

As the discovery of new materials and functionalities are occurring at an unprecedented rate, new strategies to accelerate the connection between material state or property to a desired application are necessary. Despite the availability of modern tools, the slow, tedious, and Edisonian research practices of the past are still the standard for areas such as thin films and coatings. The development of a new thin film requires either starting from a known, reported recipe, or the use intuition to determine a suitable starting condition. After thin film deposition is complete, there may be

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post-growth optimization and characterization, with many iterations of this process necessary until the initial growth conditions match some desired end state.

Laser spike annealing (LSA) is a promising technique for rapidly investigating the modification of thin film materials due to thermal annealing. By scanning a focused, continuous-wave laser across a thin film surface, localized heating can be achieved with heating/quenching times as short as microseconds and temperatures exceeding 1000°C. The laser intensity, scan rate, and annealing environment can be varied, allowing thousands of distinct annealing conditions to be studied on a single sample, including dwell times several orders of magnitude shorter than what can be achieved with rapid thermal annealing. This approach can therefore be used for accelerated materials discovery and optimization by modifying existing thin films in a localized, high throughput manner.

In this work, LSA is applied to the oxidation of various thin film materials, including transition metals such as Hf and transition metal dichalcogenides such as MoS₂. Tunable oxidation is achieved by varying the laser conditions in a controlled oxygen environment, enabling access to a wide range of effective temperatures and heating times. The resulting structural and compositional modifications are characterized using X-ray diffraction, Raman spectroscopy, ellipsometry, UV-vis absorption spectroscopy, and X-ray photoelectron spectroscopy. Applications in the study of optical and electronic materials are presented, including refractive index optimization and the development of materials for neuromorphic device applications.

4:00pm SE-ThA-8 Towards Reliable X-Ray Photoelectron Spectroscopy: Understanding Fundamental Differences between Analyses Performed on Samples with and Without Electrical Contact to Spectrometer, *Grzegorz (Greg) Greczynski*, Linköping University, Department of Physics, Sweden

X-ray photoelectron spectroscopy (XPS) analyses of insulators or inhomogeneous samples containing insulating phases are often done with specimens electrically isolated from ground and the surface potential controlled by the flood gun. While such approach offers the possibility of delivering non-distorted spectra, the consequences for the energy level alignment between sample and the spectrometer are often not considered. The present contribution highlights these aspects by discussing several, carefully selected, case studies that visualize fundamental differences between XPS analyses performed on samples with and without electrical contact to ground. Consequences for core level shifts, energy splitting between peaks from different samples, and spectra referencing are discussed in detail. It is shown that the choice of the measurement configuration (samples with vs. without contact to the spectrometer) determines to what extent binding energies of core level peaks from conducting and insulating samples (or conducting and insulating phases of the same sample) depend on the specimens' work function. Recognizing these aspects is crucial for the correct spectra referencing and interpretation of core level shifts for series of samples with varying work function.

4:15pm SE-ThA-9 Lithography-Free Fabrication of Transparent, Durable Glass Surfaces with Embedded Functional Nanostructures, *Iliyan Karadzhov*, Rubaiya Hussain, Alessia Mezzadrelli, ICFO-Institut de Ciències Fotoniques, Spain; Wageesha Senaratne, Prantik Mazumder, Corning Research and Development Corporation; Valerio Pruneri, ICFO-Institut de Ciències Fotoniques, Spain

Glass is central to displays, touchscreens, medical interfaces, and public interactive systems. Integrating additional functions such as antimicrobial activity and mechanical durability to optical clarity is of significant commercial interest which, however, remains challenging. Lithographic techniques provide excellent control over nanoscale geometry and are widely used for surface patterning. However, for applications where random subwavelength features are acceptable, lithography-free routes based on self-assembled metal masks can reduce process complexity and improve compatibility with large-area glass processing.

Among candidate antimicrobial materials, Cu offers well-established ion-mediated antibacterial activity; however, exposed Cu nanostructures are susceptible to wear and optical degradation, limiting their practical use on transparent surfaces. Embedding Cu within glass nanostructures is therefore an attractive strategy to simultaneously enable durability and sustained functionality. In our previous work on abrasion-resistant antireflective glass, thermally dewetted Ag nanoparticles were used to form a Ni nanohole mask for dry etching subwavelength nanoholes into glass. While effective for creating durable antireflective textures, this approach was not suitable for embedding Cu because a selective etchant that could

remove the Ni mask without attacking or degrading the embedded Cu could not be identified.

We overcome this limitation by replacing the Ni hard mask with a diluted polymer nanohole mask, enabling wet glass etching and subsequent lift-off of Ti/Cu nanodisks embedded below the glass surface. The resulting surfaces show 80–85% visible transmission, haze below 1%, and near-neutral color. Antimicrobial testing against *E. coli* OP50 shows approximately 99% bacterial reduction after 1 h, while crockmeter abrasion causes no measurable optical change. Future work will optimize geometry and Cu release to improve antimicrobial efficacy toward sanitizer-level benchmarks and validate performance against standard test strains.

Advanced Surface Engineering Room Ballroom A - Session SE-ThP

Advanced Surface Engineering Poster Session

SE-ThP-1 PLD or Magnetron Sputtering: Which to Choose for Functional Coatings?, *Esteban Broitman, Sven Kelling, Rickmer Kose, SENTYS*

This review evaluates pulsed-laser deposition (PLD) and magnetron sputtering (MS) through a practical, application-oriented lens to help researchers and engineers select the most appropriate route for functional thin films (oxides, nitrides, chalcogenides, carbon-based). Drawing on head-to-head studies that deposit identical materials with both techniques, the paper traces how differences in deposition physics translate into film composition, microstructure, defect populations, mechanical properties and device performance.

PLD is highlighted for its reliable stoichiometric transfer from complex targets, its ability to produce high bonding density and elevated sp^3 fractions in carbon films, and its suitability for epitaxial growth and high-hardness coatings; its principal drawback is the tendency to generate particulates from the energetic plume unless droplet-mitigation strategies are employed. Magnetron sputtering (DC/RF) is shown to excel at large-area uniformity, conformal coverage and low macroscopic droplet density; pulsed modes and HiPIMS further boost ionization and substrate bombardment, yielding dense films that can rival PLD in many functional metrics while offering established industrial throughput.

The review also surveys hybrid PLD+MS approaches that leverage PLD's compositional fidelity together with sputtering's smoother deposition to reduce defects and increase density. Advances in ultra-high vacuum (UHV) platforms enhance both methods: UHV PLD improves cleanliness and epitaxial control, and UHV MS reduces impurity uptake and tightens reproducibility. Manufacturers of scientific research equipment such as **PREVAC** exemplify systems that enable in-situ diagnostics and clean sample transfer. Finally, the paper places cost and scalability in context: bench-scale sputter tools are typically less costly to acquire and run than PLD benches, whereas at production scale capital expenditures for high-end HiPIMS sputter systems and wafer-scale PLD platforms converge.

The review closes with a concise decision matrix and actionable guidelines to match deposition choice to stoichiometry, surface quality, throughput and budget constraints.

SE-ThP-2 Surface Engineering of Ni-Based Alloy Thin-Film Catalysts for Enhanced Hydrogen Evolution Reaction, *Zhe-Yu Liu, Jih-Siang Yang, Fan-Bean Wu, Wan-Yu Wu*, National United University, Taiwan

Nickel-based thin-film catalysts have attracted increasing interest as cost-effective alternatives to noble-metal electrocatalysts for water electrolysis. In this study, Ni-Cu and Ni-Mo alloy thin films were prepared by magnetron co-sputtering as a surface engineering strategy to tune composition, morphology, and electrocatalytic performance for the hydrogen evolution reaction (HER). The sputtering process enables the fabrication of dense, adherent, and composition-controlled thin films, providing a useful platform for clarifying the relationship between alloy composition and catalytic activity. For the Ni-Cu system, Cu was incorporated into Ni to balance hydrogen adsorption behavior and improve charge-transfer properties. Pure Ni tends to bind hydrogen too strongly, whereas Cu has weaker hydrogen adsorption and high electrical conductivity. Therefore, the Ni-Cu alloy design is expected to provide a synergistic effect for HER. Electrochemical measurements in 1 M KOH showed that the Ni-Cu thin film with a near-equiatomic composition of approximately Ni:Cu = 55:45 exhibited the best HER performance, achieving overpotentials of 76 mV at 10 mA cm^{-2} and 180 mV at 100 mA cm^{-2} . This film also showed the lowest charge-transfer resistance of 0.71Ω , indicating enhanced interfacial reaction kinetics. For the Ni-Mo system, Mo-Ni alloy thin films with different Ni contents were deposited by DC co-sputtering on silicon and carbon-paper substrates. Structural and surface analyses indicated that co-sputtering promoted composition-dependent thin-film structures with hydrophilic surfaces. In $0.5 \text{ M H}_2\text{SO}_4$, the Mo-Ni catalysts exhibited a volcano-type dependence of HER activity on Ni content. The catalyst with 72.8 at% Ni showed the optimal performance, with an overpotential of 149.0 mV at 10 mA cm^{-2} and a Tafel slope of 77.5 mV dec^{-1} . In 1 M KOH, HER activity generally improved with increasing Ni content, suggesting the important role of Ni in alkaline HER kinetics. Overall, these results demonstrate that magnetron co-sputtering is an effective approach for tailoring the composition and surface properties of Ni-based alloy thin-film

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catalysts. The Ni-Cu system highlights the importance of balancing hydrogen adsorption and charge transfer, while the Ni-Mo system shows that composition-dependent structural modification strongly influences HER kinetics.

SE-ThP-5 Synthesis and Dehydrogenation of Thin Film Metal Hydride for Hydrogen Storage, *Vladimir Gorokhovskiy, Grace Owens*, University of Colorado at Boulder; *Paul Smith*, Plasma Kinetics Corporation

Magnesium hydride (MgH_2) is solid-state hydrogen storage material, with a theoretical hydrogen storage capacity of 7.6 wt%, making it an attractive candidate due to its high hydrogen affinity, low cost and abundance. But high enthalpy of formation and high decomposition energy of MgH_2 requiring about 76 kJ/mol of heat for each mole of H_2 released lead to increase of hydrogenation temperature, slow hydrogenation and dehydrogenation kinetics. Ni is often used as a catalyst to improve the kinetics of MgH_2 formation in H_2 -contained atmosphere due to reduced enthalpy of formation of Mg-Ni alloys ($<65 \text{ kJ/mol}$) and also has a reduced activation energy of dissociation of molecular hydrogen in comparison to Mg, suggesting that Ni mainly catalyzes the dissociation/recombination reaction barrier for H_2 solid-state hydrogen storage material reducing the hydrogenation/dehydrogenation temperatures. MgNi thin film coatings were prepared via vacuum cathodic arc deposition on sapphire wafer, aluminum foil, and aluminized polyamide tape. The cathodic arc process conditions for deposition of MgNi coatings were chosen to keep the substrate temperature low for which the arc current was selected from 50 to 70 A, and substrates (both sapphire and metal foil) were installed in a free-standing position at floating potential. The coating architecture consisted of the bottom supporting NiTiCu sublayer having large open columnar microstructure promotes the formation of the columnar microstructure with open voids in the MgNi layer. The Pd top layer deposited by e-beam evaporation improves hydrogen absorbance capability. The films were hydrogenated in $\text{Ar}/5\%\text{H}_2$ atmosphere at 300°C in atmospheric pressure. The hydrogenation changed the color of the coating surface from silver metal to dark color accompanied by reduced electrical conductivity and increased transparency of the film indicating shift from metallic to insulative ceramic. The dehydrogenation of the magnesium hydride containing films was made via laser irradiation at room temperature using 808 nm wavelength CW laser. The hydrogen flow, consisting of both molecular and atomic hydrogen, generated by laser irradiation of one hydrogenated coating spot $1 \text{ mm} \times 10 \text{ mm}$ during 1s ranging from 0.5 to 1 SCCM. It was demonstrated that almost 90% of hydrogen contained in hydrogenated NiTiCu/MgNi/Pd coating can be released by CW laser irradiation from the $1'' \times 1.25''$ sample. The partial light transparency and increased light absorbance of MgH_2 improved by columnar microstructure with open voids provide a basis for using amplified light beam to release hydrogen from the thin film, contributing to safe and efficient hydrogen storage.

SE-ThP-6 Photodegradable Thin Films for Dust Mitigation, *Kira Sand, David Allred*, Brigham Young University

High-contrast optical systems for space applications require effective dust mitigation strategies to maintain low-scatter surfaces and edges. A novel approach would be to use photodegradable thin films that can remove any dust particles after launch, thereby reducing the likelihood of further particulate accumulation.

In this work, we investigate the photodegradation behavior of polymers such as poly(olefin sulfone) (PMPS) and poly(1-hexene sulfone) (PHS) under space-like conditions, specifically in vacuum under UVC irradiation (254 nm and 172 nm). We demonstrate that these materials undergo efficient dissociative degradation, enabling the removal of dust from coated optical surfaces. Our results show a significant reduction in particle coverage following irradiation of dust-coated films.

Additionally, we report the use of more energetic thin-film materials, including ammonium nitrate, which exhibit similar photodegradation behavior. These materials may provide enhanced dust removal efficiency due to the higher energy stored in their chemical bonds.

Overall, this study highlights the potential of photodegradable thin films as an effective strategy for in situ dust mitigation in space-based optical systems.

SE-ThP-7 Surface Patterning with Unconventional Self-Assemblies by an *m*-Terphenyl Isocyanide Ligand, *Liya Bi, Zhiyuan Yin, Krista Balto, Joshua Figueroa, Shaowei Li*, University of California San Diego

Self-assembled monolayers (SAMs) refer to spontaneously formed single-layer molecular sheets on surfaces, which have found applications in

corrosion protection, catalysis, drug delivery, molecular sensing, etc. The assembling behaviors of a given molecule on the surface depend on both the molecule-surface interactions and the intermolecular interactions which include covalent bonding, van der Waals interaction, hydrogen bonding or a combination of these forces. Consequently, there are various tuning knobs towards controllable molecular self-assemblies by modifying either the surfaces or the molecules. Here, we report the unconventional self-assembled patterns of a rationally designed *m*-terphenyl isocyanide ligand, CNAr^{Mes2}, on Ag surfaces. This molecular ligand binds to metal atoms via its isocyanide group and experiences rich intermolecular interactions upon surface adsorption because of its *m*-terphenyl skeleton. We anticipated it to form a dimerized linear molecular chain on metal surfaces based on its profile, as confirmed on Cu(100) and Cu(111) surface. Surprisingly, CNAr^{Mes2} self-assembles into a few unexpected structures on Ag(111) surface including ribbons, rings and hexagons at low surface coverage and forms closely packed monolayers at high molecule density. A detailed study on these unconventional patterns suggests that CNAr^{Mes2} trimer other than the dimer is the building block and that the surface chemistry of Ag plays a vital role in this assembling mechanism change. These observations inspire us to engineer the metal-binding isocyanide group and/or the steric bulks of CNAr^{Mes2} to achieve programmed SAMs with on-demand shapes, lattice constants and functionalities on metal surfaces.

SE-ThP-9 Heterointerface-Induced Charge Transfer Kinetics in Sputtered MoTe₂-MoS₂ Nanocomposite for High-Performance Ambient NO₂ Detection, Sonika Kodan, **Ramesh Chandra**, Indian Institute of Technology Roorkee, India

The present research reports a nanostructured MoTe₂-MoS₂ n-n heterojunction-based gas sensor developed on a silicon (Si) substrate, demonstrating room-temperature (RT, 30 °C) nitrogen dioxide (NO₂) sensing. A controlled and one-step magnetron co-sputtering approach is utilized to deposit MoTe₂-MoS₂ nanocomposite thin film on a Si substrate, producing a uniform, dense, and porous nanoscale morphology. Herein, surface analysis and structural characterization of the MoTe₂-MoS₂/Si sensor reveal a rough, and defect-rich morphology with abundant exposed edge sites, which significantly enhances the availability of active adsorption sites for effective gas sensing. The developed MoTe₂-MoS₂/Si heterostructured sensor delivers an exceptional sensor response of 74.4% with an ultrafast response/recovery time of 7.5/2 s towards 25 ppm NO₂ at RT. Additionally, the present sensor showcases highly reproducible and consistent behavior over a wide range of NO₂ concentrations (1-60 ppm), along with remarkable selectivity against common potential interfering gases (H₂S, NH₃, CO, H₂) and a low detection limit of 700 ppb. This study provides critical insights into NO₂ sensing induced by heterointerface band modulation and defect-assisted adsorption at the sensor's surface, thereby providing a strong foundation for a reliable platform for high-performance NO₂ gas sensors based on the MoTe₂-MoS₂ heterostructure.

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