

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.

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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.

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