## Plasma and Vapor Deposition Processes
## Room Town & Country A - Session PP1-1-MoM

### PVD Coatings and Technologies I

**Moderator: Christian Kalscheuer**, IOT, RWTH Aachen, Germany

10:00am **PP1-1-MoM-1 Optimizing Sputter-Deposited MoS2 Coatings: Insights from Monte Carlo Simulations and In-Situ Plasma Diagnostics***, **Alexander Mings [ajmings@sandia.gov]**, Steven Larson, Kyle Doorman, Tomas Babuska, John Curry, Remi Dingreville, David Adams,* Sandia National Laboratories, USA

Sputter-deposited molybdenum disulfide ($MoS_2$) coatings are widely utilized in aerospace applications, primarily due to their exceptional reliability and ultralow friction in vacuum environments. However, the unique structure of $MoS_2$ often leads to high porosity in sputtered coatings which can significantly compromise wear life and accelerate film oxidation. To address these challenges, engineers typically engage in costly process development, varying deposition parameters to maximize film density with limited available heuristics. This process development is also equipment specific and must be repeated when a process needs to be transferred.

This study explores how the kinetic energy of species impacting the substrate during the growth of $MoS_2$ films influences their porosity. We employ Monte Carlo simulations using both SRIM (Stopping and Range of Ions in Matter) and SIMTRA (Simulating the Transport of Atoms from the Source to the Substrate) to analyze the contributions of both sputtered atoms and backscattered neutrals. By correlating these findings with nanoindentation hardness, we gain insights into how deposition dynamics affect coating porosity. Additionally, we compare the simulation results to in-situ measurements made with a Retarding Field Energy Analyzer (RFEA) positioned at the substrate. Our findings reveal the energy flux necessary to produce dense coatings, which can be used in combination with a RFEA to provide essential feedback for process development. This approach has the potential to both greatly accelerate process transfer and enhance the film performance of existing $MoS_2$ processes.

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

10:20am **PP1-1-MoM-2 HiPIMS and Digitalization: Increasing Efficiency in Machining***, **Stephan Bolz [stephan.bolz@cemecon.de]**, Biljana Mesic, Oliver Lemmer, Christoph Feig,* CemeCon AG, Germany

The presentation "HiPIMS and Digitalization: Increasing Efficiency in Machining" shows how modern digitalization and innovative coating technologies accelerate the development of HiPIMS coatings for cutting tools.

The focus is on using a HiPIMS coating system “CC800 HiPIMS,” for which a digital twin model exists that captures and analyzes large amounts of data.

Machine data are first collected via an OPC-UA interface, persisted in a database, and then transferred into the digital twin. This allows simulation results to be quickly integrated into process development. Consequently, only a few validation experiments are needed, saving time and resources. The simulation-optimized HiPIMS process parameters enable the deposition of coatings that are denser and harder than ever before. These were tested on cutting tools and achieved better results than previous layers.

Overall, the talk demonstrates how the combination of HiPIMS technology and digitalization increases efficiency in coating development, improves product quality, and strengthens competitiveness in high-performance machining.

10:40am **PP1-1-MoM-3 From Poisoned Targets to Healthy Models: The Quest for Parameters***, **Diederik Depla [Diederik.Depla@ugent.be]**,* Ghent University, Belgium **INVITED**

The conceptual simplicity of reactive magnetron sputtering facilitates the description of global trends in process curves characteristic of reactive magnetron sputtering. However, achieving a quantitative description of these trends through simulations remains far more challenging, as the critical bottleneck of every modelling effort lies in the determination of accurate input parameters. Following a brief introduction to the RSD model, this paper provides an overview of several experimental methodologies designed to extract the parameters essential for its implementation. A central parameter in any thin-film deposition technique is the deposition rate. While its determination in metallic mode is relatively straightforward, the task becomes substantially more complex in poisoned mode due to the limited availability of sputter yield data for oxides. Our experiments reveal that in poisoned mode sputter yields exhibit a pronounced dependence on process conditions. Monte Carlo simulations, moreover, uncover a remarkable material-independent correlation between reported partial yields for oxides and experimentally measured yields in poisoned mode. Another crucial quantity, the ion-induced electron yield, can only be reliably determined experimentally, even for metals. By employing empirical scaling laws, however, it becomes feasible to estimate these yields under poisoned-mode conditions. The strong influence of chemisorption on the electron yield explains the discharge voltage behaviour in metallic mode. The influence of chemisorption on target poisoning emerges as the next major challenge, particularly as a novel strategy to control the reactive sputtering process exposes discrepancies between the current formulation of the model and experimental observations. Nevertheless, this measuring strategy provides compelling evidence that the RSD model’s prediction of double hysteresis behaviour is fundamentally correct.

11:20am **PP1-1-MoM-5 Advanced YSZ Coatings Deposited by Magnetron Sputtering for High-Temperature Applications***, **Imene Toumi [imene.toumi@utt.fr]**,* Université de Technologie de Troyes, France*; Sofiane Achache,* Université de technologie de Troyes, France*; Akram Alhussein, Benoit Panicaud,* Université de Technologie de Troyes, France

Thermal barrier coatings (TBCs) are essential for extending the lifetime and efficiency of components exposed to extreme thermal environments, particularly in aerospace and energy systems [1-2]. Yttria-stabilized zirconia (YSZ) remains the benchmark topcoat material due to its low thermal conductivity, high fracture toughness, and outstanding thermal stability [1-4]. The performance of these coatings is strongly governed by the stabilization of the metastable tetragonal phase, which depends on both yttria content and deposition conditions [5].

In this study, YSZ thin films were deposited by dual-target reactive magnetron sputtering (Zr + Y) under an $Ar/O_2$ atmosphere. A parametric analysis was conducted to evaluate the effects of oxygen flow rate, substrate position, scanning amplitude, and yttrium doping level (expressed as $Y_2O_3$ content) on film growth, chemical composition, and crystalline structure.

Physicochemical and mechanical characterizations were performed using X-ray diffraction (XRD), energy-dispersive spectroscopy (EDS), scanning electron microscopy (SEM), and nanoindentation. The optimal yttria content ($Y_2O_3$) was found to be ≈ 2.9 mol%, ensuring stable tetragonal phase formation.

Furthermore, the thermal stability of the prepared coatings was assessed through annealing cycles at 500 °C, 750 °C, and 1200 °C. The study aims to evaluate the stability of the tetragonal phase at elevated temperatures and to investigate the microstructural evolution of YSZ thin films.

This work establishes a multi-parameter optimization strategy for YSZ thin films, contributing to the design of next-generation TBCs with improved structural integrity and reliability in harsh service conditions.

11:40am **PP1-1-MoM-6 Low-Temperature Synthesis of $Ti_2AC$ (A = Si or Ge) Max-Based Coatings via Highly Ionized Growth Techniques***, Arno Gitschthaler, Philipp Dörflinger, Rainer Hahn,* Christian Doppler Laboratory for Surface Engineering of high-performance Components, TU Wien, Austria*; Jürgen Ramm, Klaus Böbel,* Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein*; Szilard Kolozsvári, Peter Polcik,* Plansee Composite Materials GmbH, Germany*; Eleni Ntemou, Daniel Primetzhofer,* Department of Physics and Astronomy, Uppsala University, Sweden*; Dominik Fuchs, Andreas Limbeck,* Institute of Chemical Technologies and Analytics, TU Wien, Austria*; Peter Švec,* Institute of Physics, Slovak Academy of Sciences, Slovakia*; Anton Davydok, Christina Krywka,* Institute of Materials Physics, Helmholtz Zentrum Hereon, Germany*; **Helmut Riedl [helmut.riedl@tuwien.ac.at]**,* Institute of Materials Science and Technology, TU Wien, Austria

MAX phases are a unique class of nanolaminated compounds that combine metallic and ceramic properties, offering excellent electrical and thermal conductivity together with remarkable resistance to creep, oxidation, and corrosion. These characteristics make them highly attractive as protective and functional coatings for next-generation hydrogen technologies. However, conventional sputtering techniques struggle to provide suitable growth conditions at reduced synthesis temperatures, often leading to phase instability and the formation of competing phases. Despite more than two decades of research on Ti–A–C (A = Si or Ge) MAX coatings [1,2], it has yet to be achieved to deposit them under less harsh, more practical conditions. To address this issue, $Ti_2$-A-C (A = Si or Ge) thin films were deposited by cathodic arc evaporation (CAE) and high-power impulse magnetron sputtering (HiPIMS) of metallic TiA (A = Si or Ge) targets in reactive $Ar/C_2H_2$ plasma atmospheres. To understand the relationship between deposition parameters, chemical composition, and phase formation, the resulting films were comprehensively characterized using high-resolution techniques, including ToF-ERDA-calibrated GD-OES, 2D-BBXRD, and t-CSXRD measurements. Subsequently, these results are correlated with application near electrochemical tests. Overall, these analyses demonstrate, for the first time, that $Ti_2$-A-C MAX-based coatings can be successfully synthesized by reactive CAE and HIPIMS at temperatures as low as 550 °C and rise their potential for use cases in hydrogen technologies.

[1] Emmerlich J, Palmquist J-P, Högberg H, Molina-Aldareguia JM, Hultman L. Growth of Ti3SiC2 Thin Films by Elemental Target Magnetron Sputtering. J Appl Phys. 2004;96: 4817. doi:10.1063/1.1790571

[2] Högberg H, Eklund P, Emmerlich J, Birch J, Hultman L. Epitaxial Ti2GeC, Ti3GeC2, and Ti4GeC3 MAX-phase thin films grown by magnetron sputtering. J Mater Res. 2005;20: 779–782. doi:10.1557/JMR.2005.0105

12:00pm **PP1-1-MoM-7 Development of a Thickness Ratio Design Model for TiN/Ti/TiN/Ti Coating: Stress and Energy Relief Analysis***, **Yi-Cheng Yang [richardyang329@gmail.com]**,* Department of Engineering and System Science National Tsing Hua University, Taiwan

Transition metal nitride (TMeN) coatings deposited by physical vapor deposition (PVD) are often subjected to high residual stress, which may induce delamination and failure of coatings. Introducing a metal interlayer between the coating and substrate is one of the common approach to relieve residual stress. However, the design of interlayer thickness in commercial applications generally lacks quantitative evaluation. In our previous studies, we developed an energy-balance model to quantitatively evaluate the stress and energy relief efficiency of coatings using ZrN/Ti, ZrN/Zr and TiN/Ti as model systems. With the growing demand for multilayer structures, optimizing the thickness ratio between coating and the metal interlayer for effective stress and energy relief has become essential for improving coating performance and process efficiency. Therefore, this study aims to evaluate the thickness ratio between coating and interlayer using TiN/Ti/TiN/Ti as a model system. By employing the energy-balance model under bilayer conditions, the effect of varying Ti and TiN layer thickness ratios on stress and energy relief behavior will be comprehensively analyzed. In this study, TiN coatings were deposited on Si substrates using DC unbalanced magnetron sputtering (DC-UBMS). Residual stress was measured by laser curvature measurement and the average X-ray strain (AXS) method. Based on the optimized model, the influence of thickness ratio on the stress and energy relief in multi-layer structures can be quantitatively evaluated.

## Plasma and Vapor Deposition Processes
## Room Town & Country A - Session PP1-2-MoA

### PVD Coatings and Technologies II

**Moderators: Yen-Yu Chen**, National Pingtung University of Science and Technology, Taiwan , **Christian Kalscheuer**, IOT, RWTH Aachen, Germany

1:40pm **PP1-2-MoA-1 Thick Uniform Coatings on Hollow Glass Microspheres via DC Magnetron Sputtering***, **Kira Shulman [kirashulman@lanl.gov]**,* Los Alamos National Laboratory, USA

While DC magnetron sputtering is excellent for coating planar substrates, coating spherical substrates is challenging due to the line-of-sight nature of this deposition process. To address this limitation, a method of agitation must be employed to keep these spherical substrates “rolling”, thereby exposing all substrate surfaces to the sputter source throughout the coating process. Furthermore, producing thick coatings on microscopic spherical substrates is problematic due to issues balancing production of larger batches with coating consistency. In this work, we develop a vibrating stage assembly to agitate hollow glass microspheres (HGMS) of
varying diameters and optimize coating parameters in order to produce thick uniform coatings via magnetron sputtering. The vibrating stage assembly provides the linear mechanical agitation necessary to allow for optimal intermixing of the HGMS, resulting in increased uniformity of the coatings. To address thickness concerns, we found that running coatings for longer periods of time as well as minimizing the distance from sputter source to substrate ensured optimal development of thicker coatings on smaller batches of HGMS. An eleven hour coating run with source-substrate distance at 6 inches produced 120 nm vanadium coatings on 25-28 um HGMS, while a ten hour coating run with source-substrate distance at 3.5 inches resulted in 300 nm tungsten coatings on 32-38 um HGMS. Analysis of this data confirms that linear agitation and fine-tuning sputter coating parameters does support uniformity and ensures thick coatings of HGMS. While these are promising results, this system can only maintain high uniformity and thickness when coating small batches of HGMS at a time. Efforts to develop a larger system capable of producing much higher yield without comprising uniformity and thickness will be discussed.

2:00pm **PP1-2-MoA-2 Investigation of the Relationship Between Film Texture and Hydrogen Barrier Characteristics in HCD-IP ZrN Thin Films on Zircaloy-4***, **Cheng-Han Wu [Jordan91618@gmail.com]**, Kuan-Che Lan,* National Tsing Hua University, Taiwan

The mechanical integrity of Zircaloy-4 claddings, widely used in light-water reactors, is essential for maintaining structural reliability and preventing hydrogen-induced degradation. To mitigate hydrogen ingress, protective zirconium nitride (ZrN) coatings were deposited on Zircaloy-4 substrates using a hollow cathode discharge-ion plating (HCD-IP) system. This study aims to explore how crystallographic texture may affect the hydrogen barrier characteristics of the ZrN films. By adjusting the oxygen flow ratio during deposition, distinct texture evolution from (111) to (200) orientations was achieved. SEM and FIB will focus on the surface morphology and hydride layer formation, while XRD and GIXRD will be employed for phase identification, grain size estimation, and residual stress evaluation. EPMA and XPS measurement will include the nitrogen-plus-oxygen-to-zirconium ratio (N+O/Zr ratio) and depth profile analysis to clarify the compositional variation with oxygen addition. The potential relationship between preferred orientation and hydrogen resistance will be investigated to discuss whether texture control in HCD-IP deposition plays a role in the hydrogen barrier characteristics of ZrN coatings.

2:20pm **PP1-2-MoA-3 Spherical Tungsten Coating as Inertial Fusion Targets***, **Ali Basaran [ali.basaran@ga.com]**, Priya Raman, Pavel Lapa, Ruben Santana, Hongwei Xu, Wendi Sweet, Fred Elsner, carlos monton,* General Atomics, USA*; Sasikumar Palaniyappan, Eric Loomis,* Los Alamos National Laboratory, USA

High density uniform tungsten coating on microscale spherical shells is of great interest for next generation inertial fusion energy targets since high-z shells are proposed to improve ablator performance and resistance to preheating. In this work, we present deposition of tungsten films on polymeric shells using direct current magnetron sputtering. Spherical shells are agitated on a pan to ensure uniform coverage during deposition. Delamination of high-z metals from the pan during thick coatings and agitation patterns that determine the surface finish of shells are addressed through several strategies. Process parameters such as pressure, power, and target-substrate geometry are optimized to achieve dense coatings with thicknesses up to 50 μm while minimizing residual stress, roughness, and porosity. Metrology of the shells such as thickness, sphericity, and roughness are quantified via x-ray, optical, and electron microscopy techniques. The influence of deposition conditions on coating microstructure and surface morphology will be discussed.

Acknowledgement

This material is based upon work supported by the U.S. Department of Energy, Office of Science, Office of Fusion Energy Sciences, under award number DOE/NNSA award 89233124CNA000365.

## Plasma and Vapor Deposition Processes
## Room Town & Country B - Session PP2-1-WeM

### HIPIMS, Pulsed Plasmas, and Energetic Deposition I

**Moderators: Arutiun P. Ehiasarian**, Sheffield Hallam University, UK, **Tetsushide Shimizu**, Tokyo Metropolitan University, Japan

8:00am **PP2-1-WeM-1 Alpha-alumina thin films at low temperature: how R-HiPIMS process parameters influence purity and crystallinity***, **Célia Dieudonné [celia.dieudonne@icmcb.cnrs.fr]**,* ICMCB, France*; Marjorie Cavarroc-Weimer,* Safran, France

Reactive High Power Impulse Magnetron Sputtering (R-HiPIMS) is emerging as a highly effective technique for the deposition of high-quality insulating metal oxide coatings. There are numerous process parameters. Some are similar to those of reactive conventional magnetron sputtering (working pressure, gas ratio, substrate temperature etc.), while others are similar to those of pulse use (frequency, time-off etc.). This study highlights the potential of R-HiPIMS to produce dense, homogeneous, and pure alpha-alumina ($\alpha$-$Al_2O_3$). High ionization rates and peak power densities inherent to R-HiPIMS play a crucial role in promoting film densification and stabilizing the $\alpha$-$Al_2O_3$ phase. The influence of other process parameters including oxygen partial pressure, working pressure, target power density, and substrate temperature on film microstructure is investigated. Special attention is given to the effect of the bias configuration of the steel substrate, comparing the presence of $\alpha$-$Al_2O_3$ when the substrate is grounded or at a floating potential. This aspect constitutes a major focus of the study, as the substrate bias state significantly affects the energy and flux of incoming ions, as well as the overall plasma–substrate interaction during deposition. A mechanism to stabilize $\alpha$-$Al_2O_3$ is proposed.

8:20am **PP2-1-WeM-2 Influence of Pulse Parameters in Multi-Pulse Hipims on Reactive Mode Transition for $VO_2$ Thin Film Deposition***, **Erdong Chen [chen-erdong@ed.tmu.ac.jp]**, Rina Watabe,* Tokyo Metropolitan University, Japan*; Stephanos Konstantinidis,* University of Mons, Belgiums, Belgium*; Daniel Lundin,* Linköping University, Sweden*; Tetsuhide Shimizu,* Tokyo Metropolitan University, Japan

Vanadium dioxide (VO2) undergoes a thermochromic phase transition around 68°C, changing from a tetragonal to a monoclinic structure accompanied by a substantial change in optical and electrical properties, which can be used in e.g., smart windows. However, synthesizing single-phase VO2 films via reactive sputtering remains challenging due to 1) the wide range of vanadium-oxygen (V-O) stoichiometries, and 2) the need to grow the proper crystalline phase, leading to a limited process window with a very narrow phase transition from VOx to $VO_2$.

In this study, these challenges were addressed by regulating the peak current (Ipk) in reactive multi-pulse High-Power Impulse Magnetron Sputtering (HiPIMS) to control the reactive mode transition. The number and on-time of micro-pulses were systematically varied to elucidate the relationship between the incident ion flux and Ipk evolution as a function of $O_2$ gas flow, using ion mass spectrometry.

With this approach, a linear increase in Ipk was achieved upon $O_2$ introduction, mitigating the abrupt current drop and reducing process hysteresis typically observed in single-pulse HiPIMS. This enabled more stable control over $VO_2$ deposition and improved the deposition rate within the transition regime.

As a result, $VO_2$ crystalline thin films were successfully deposited on ZnO/glass substrates. Thermochromic measurements revealed a resistivity change of over two orders of magnitude across the phase transition occurring at approximately 50 °C.

8:40am **PP2-1-WeM-3 Bipolar HiPIMS Discharges: Principles, Diagnosticsand Thin Film Deposition Strategies***, **Jiří Čapek [jcapek@kfy.zcu.cz]**, Tomáš Kozák, Andrea Dagmar Pajdarová, Mina Farahani, Tomáš Tölg,* University of West Bohemia in Pilsen, Czechia **INVITED**

The properties of thin films depend on their microstructure, crystal structure, and residual stress, which are influenced by the mobility of adatoms during growth. In magnetron sputtering, the adatom mobility can be enhanced via ion bombardment by applying a bias voltage. High-power impulse magnetron sputtering (HiPIMS) offers greater control by delivering high-power density pulses, producing a high fraction of ionized species and enhanced ion bombardment, even without a substrate bias. However, a bias voltage may still be needed to control low-energy ions. Recently, bipolar HiPIMS, where a positive voltage pulse follows the main negative voltage pulse, has been suggested to be used instead of a substrate bias voltage.

This presentation summarizes our research on bipolar HiPIMS. Plasma analyses using the Langmuir probe and mass spectroscopy revealed that plasma parameters evolve similarly regardless of positive pulse parameters or distance from the target, though their values differ. During the initial phase of the positive pulse, a large potential difference (up to 200 V), high electron temperature (up to 150 eV), and a significant drop in electron density were observed. After this part, the difference between the potentials and the electron temperature is low. The time-averaged spectra of ions exhibit a prominent high-energy peak. It is shown that the position of the peak can be varied by the positive pulse amplitude, its magnitude scales with the pulse length, and its width can be slightly influenced by the length of the delay interval.

Special attention is devoted to the deposition of films on insulating surfaces. Biasing such surfaces becomes ineffective because the plasma-substrate potential difference necessary for ion acceleration almost vanishes once the surface is charged by the incident plasma ions. We propose the utilization of chopped bipolar HiPIMS (featuring several short positive pulses replacing a single long positive pulse) to enhance energy flux to such insulating surfaces. Results show that for an insulated surface with low capacitance, bipolar pulse configurations do not significantly increase energy flux to the surface due to its rapid charging by plasma ions. Conversely, high surface capacitance facilitates an increase in energy flux even for a long positive pulse. For medium surface capacitance (tens of nF), chopping the positive pulse in bipolar HiPIMS effectively increases the energy delivered to the film by discharging the surface in the off-times. Optimal conditions for the deposition of thin films will be discussed based on this systematic study of various unipolar and bipolar pulse configurations.

9:20am **PP2-1-WeM-5 Synthesis-Dependent Phase Evolution in the W–N System: A Case Study with HiPIMS and N+ ion source***, **Oleksandr Pshyk [oleksandr.pshyk@empa.ch]**, Kerstin Thorwarth, Nathan Rodkey, Sebastian Siol,* Empa - Swiss Federal Laboratories for Materials Science and Technology, Switzerland

Many nitride compounds exhibit relatively wide homogeneity ranges, which usually simplify their synthesis. However, some complex nitrides, such as W-N, contain numerous stable ground-state phases as well as metastable phases. Some of these phases have been computationally predicted to possess extraordinary properties, especially the nitrogen-rich compounds. In contrast, some of these phases have a very narrow homogeneity range, which, combined with variations in synthesis conditions, complicates their controlled synthesis. Exploring such a complex phase space becomes even more challenging when non-equilibrium synthesis methods, such as physical vapor deposition (PVD) methods, are employed. Therefore, understanding the phase evolution sequences within the W-N system under the exceptional synthesis conditions provided by high power impulse magnetron sputtering (HiPIMS) can help identify the conditions necessary for the synthesis of unique nitride compounds.

In this report, we present a comprehensive exploration of the basic synthesis parameter space for W-N thin films using HiPIMS. In addition to varying the nitrogen partial pressure (and thus the nitrogen-to-tungsten ratio), we investigate the effects of substrate temperature, substrate bias potential, and substrate-to-target distance on phase formation. We show how the phase fractions within each composition window change with variations in these process parameters. Furthermore, to elucidate the role of $N_2$ gas dissociation and ionization on phase evolution, we perform W-N thin film deposition assisted by an electron cyclotron wave resonance nitrogen plasma source. Our results reveal that the synthesizability of the two most commonly reported W-N phases synthesized using PVD methods - the NaCl-structured $WN_x$ and WC-structured $WN_x$ phases - strongly depends on the nitrogen concentration in the films set by substrate temperature or substrate bias. Furthermore, the boundaries between different phases are highly sensitive to the deposition rate, which is determined by the substrate-to-target distance. We analyze and discuss these results in the context of plasma characteristics at different nitrogen partial pressures and substrate-to-target distances.

The findings presented here can serve as a guide for synthesizing other compounds within complex phase diagrams that contain numerous stable and metastable phases within narrow homogeneity ranges.

9:40am **PP2-1-WeM-6 Development and Optimization of CrN and CrSiN Hipims Coatings for Enhanced Tool Performance in Cryogenic Machining of Ti6Al4V**, ***Gaya CHETTOUH [gaya.chettouh@utt.fr]***, University of Technology of Troyes (UTT), France; *Soufyane ACHACHE, Lamine GUEYE,* Université de Technologie de Troyes, France; *Yoann PINOT,* École Supérieure Nationale d'Arts et Métiers de Cluny, France; *Frederic SANCHETTE, Mohamed EL GARAH,* Université de Technologie de Troyes, France; *Corinne NOUVEAU,* École Supérieure Nationale d'Arts et Métiers de Cluny, France

Cryogenic machining is a sustainable alternative to conventional cutting fluids, reducing environmental impact while improving cooling efficiency at the tool–workpiece interface. This study develops protective coatings for cutting tools used in the cryogenic machining of Ti6Al4V titanium alloy. Although Ti6Al4V offers excellent mechanical strength and corrosion resistance, its poor machinability due to low thermal conductivity, high hardness at elevated temperatures, and strong chemical reactivity remains challenging.

Coatings are commonly applied to tungsten carbide tools to enhance their mechanical and tribological behavior. Lin et al. [1] showed that CrN deposited by modulated pulsed power magnetron sputtering (MPPMS) reached a hardness of 26 GPa and a Young's modulus of 330 GPa, while Si addition (6.3 at. %) by pulsed DC sputtering increased hardness and modulus to 38 GPa and 395 GPa, respectively. This work reports on the enhancement of CrN and CrSiN coatings deposited by high power impulse magnetron sputtering (HiPIMS). Deposition parameters, including nitrogen flow, target duty cycle, bias voltage, and bias pulse synchronization ($T_{on}$)—were optimized to improve film adhesion and density.

Microstructural and phase analyses were carried out using X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive spectroscopy (EDS). Nanoindentation revealed a maximum hardness of 33.2 GPa and modulus of 317 GPa at $T_{on}$ = 110 µs. Tribological tests with a rotary tribometer showed friction coefficients between 0.51–0.53 against Ti6Al4V balls, with a minimum wear volume of 1.24 × $10^{-5}$ $mm^3$/Nm at $T_{on}$ = 210 µs. Coatings deposited at $T_{on}$ = 210 µs and 310 µs also exhibited superior adhesion, with critical loads ($LC_3$) of 4.5 N and 4.15 N, respectively. The CrN coating at $T_{on}$ = 210 µs offered the best balance between mechanical and tribological performance, highlighting the key role of bias pulse synchronization in HiPIMS coatings.

Finally, the effect of Si incorporation was studied using a hybrid HiPIMS/pulsed DC mode to deposit CrSiN coatings containing 0–10 at. % Si. The influence of Si on microstructure and mechanical properties was compared with the optimized CrN reference. Coatings were then tested under cryogenic conditions to assess their machining performance. The corresponding results are presented.

[1] Lin et al. Surf. Coat. Technol., vol. 216, p. 251–258, 2013

11:00am **PP2-1-WeM-10 Nanopatterned Nanolayer TiN/NbN Coatings as Plasmonic and Wear Resistant Antimicrobial Materials**, ***Arutiun P. EHIASARIAN [a.ehiasarian@shu.ac.uk]***, *Arunprabhu Arunachalam Sugumaran,* Sheffield Hallam University, UK; *Ryan Bower, Ming Fu,* Imperial College London, UK; *David Owen, Ethan Muir, Yashodhan Purandare, Papken Ehiasar Hovsepian,* Sheffield Hallam University, UK; *Peter K. Petrov, Rupert Oulton,* Imperial College London, UK; *Thomas Smith,* Sheffield Hallam University, UK

Light-activated antimicrobial materials based on superhard nanoscale multilayer coatings are a novel class of materials which avoid eluting toxic heavy metals, are activated by visible light and are resistant to wear. High Power Impulse Magnetron Sputtering (HIPIMS) has been used to fabricate TiN / NbN nanoscale multilayers, whose surface was patterned into a nanoscale spike array using reactive ion etching to create features that amplify light-induced surface plasmon resonance. The film bilayer thickness was tailored and graded to enhance the toughness of the nanospikes and improve their resistance to sliding wear as evaluated in pin-on-disk tests and SEM observations. A highly activated plasma chemistry was observed in the HIPIMS environment with metal-to-gas ion momentum ratios reaching 4 for NbN and 2.7 for TiN at pulse duration of 200 µs. These conditions stabilised a (200) crystallographic texture for the nanolayer stacks. A high density microstructure observed in AFM curbed plasmon losses by reducing the density of scattering centres at grain boundaries. Preferential oxidation of NbN on the surface detected through XPS was responsible for deteriorating the plasmonic figure of merit of the films as observed through ellipsometry. Pump-probe laser measurements showed significant increases in the lifetime of active electron species in the films due to trapping of hot carriers by oxygen vacancies such as $Nb^{3+}$ and $Ti^{3+}$, with Nb being more sensitive due to a higher enthalpy of its oxide. An enhanced Raman scattering was observed from nanospike regions. Antimicrobial activity of up to 4-log kill was observed *for Staphylococcus aureus* and *Pseudomonas aeruginosa* bacteria under UV illumination.

11:20am **PP2-1-WeM-11 Nitride thin films and R-HiPIMS parameter selection: a machine learning approach**, ***Julien Neyrat [julien.neyrat@safrangroup.com]***, *Marjorie Cavarroc-Weimer, Edern Menou, Thomas Vaubois,* Safran, France; *Dominique Michau, Angéline Poulon-Quintin,* CNRS-ICMCB, France

Reactive High-Power Impulse Magnetron Sputtering (R-HiPIMS) is a very efficient process to elaborate high quality thin films (dense, continuous, smooth, homogeneous and nanostructured) thanks to a high-ionized plasma and to high peak power densities applied to the metallic target during short pulses. However, R-HiPIMS involves numerous parameters. Some are similar to reactive conventional magnetron sputtering (working pressure, ratio of gazes, substrate temperature...), others are specific to R-HiPIMS with the use of pulses (frequency, duration ...). This is why a new approach combining traditional knowledge-based approaches with data-driven methodologies that require artificial intelligence and machine learning algorithms is proposed. First, the links existing between film microstructures and R-HiPIMS parameters, based on design of experiments, is introduced for vanadium nitride thin films selected for tribological applications. Their excellent lubrication ability at high temperature with the formation of self-lubricant oxides, makes them excellent candidates. Second, a numerical approach is introduced to facilitate/accelerate the R-HiPIMS parameter selection. Based on the previous experimental data base, using machine-learning tools, different classification and regression models are trained. The aim is to show how this approach could be useful to predict film microstructure from process parameters and then, tribological properties.

11:40am **PP2-1-WeM-12 Effect of Coating on the Crystal Structure and Properties of VN Coatings Deposited by HiPIMS**, ***Te-I Huang [dd113011513@gapp.nthu.edu.tw]***, Department of Engineering and System Science National Tsing Hua University, Taiwan

Vanadium nitride (VN) is one of the promising coating materials for cutting tools due to its excellent mechanical properties such as high hardness and low wear rate; especially the formation of Magnéli-phase oxides at high temperature can significantly lower the friction coefficient and increase the wear resistance. In our previous study, we found that the mechanical properties and thermal stability of VN coatings were influenced by the crystal structure, where VN phase with hexagonal crystal structure (h-VN) exhibits higher hardness and better oxidation resistance compared with cubic VN (c-VN) phase. Although VN with mixed cubic and hexagonal phases were produced, the process parameters that could enhance the transition from c-VN to h-VN remain uncertain. In this study, VN coatings having different c-VN/h-VN ratios with thickness ranging from 500 to 1750 nm were deposited on Si substrates using high-power impulse magnetron sputtering (HiPIMS). The phase evolution and microstructure of the VN coatings were examined using X-ray diffraction (XRD) and scanning electron microscopy, respectively. The distribution of c-VN and h-VN phases in the VN coatings were observed using transmission electron microscopy. Residual stress of the coatings was determined using laser curvature method and XRD. The variation of residual stress, hardness, and electrical resistivity were correlated to the crystal structure of the VN coatings. The results reveal that increasing coating thickness may promote the transition from c-VN to h-VN, thereby affecting the mechanical performance of the VN coatings.

## Plasma and Vapor Deposition Processes
## Room Town & Country B - Session PP2-2-WeA

### HiPIMS, Pulsed Plasmas, and Energetic Deposition II

**Moderators: Arutiun P. Ehiasarian**, Sheffield Hallam University, UK, **Tetsushide Shimizu**, Tokyo Metropolitan University, Japan

2:00pm **PP2-2-WeA-1 Understanding the Hyper-Power Impulse Magnetron Discharge and related Arc Transition**, ***Tiberiu Minea* [tiberiu.minea@universite-paris-saclay.fr]**, *Erwan Morel, Zakaria Belkaid, Adrien Revel,* University of Paris-Saclay, France **INVITED**

High-power impulse Magnetron Sputtering (HiPIMS) has already proven its advantages over conventional magnetron discharge. Using refractory metals or graphite as target materials paved the way for much higher current densities in HiPIMS exceeding 10 A $cm^{-2}$ [1]. In addition, replacing the argon with helium leads to even higher currents, despite the discharge transition from glow to arc, under certain conditions.

The experimental findings suggested the crucial role played by self-sputtering at high voltage (~1 kV) and gas recycling at a lower pulse voltage [2]. Recent global modeling proved this scenario and unveiled microscopic information on the He/Mo HiPIMS discharge [3]. A cross-correlation with a high-speed gated camera and optical emission spectroscopy measurements revealed the plasma evolution [2]. The electron density is highly dependent on the presence of metal vapor in the plasma.

Gas preionization (low DC current) significantly improves the current rise to 1 kA when a high voltage pulse is applied, even for long pulses of 1,000 µs. Five times more power can be transferred into the plasma compared to the HiPIMS without preionization. Consequently, this operation mode has been referred to as the Hyper-Power Impulse Magnetron (HyPIM) [4]. The metastable gas states effectively sustain this high-density plasma in glow mode [5].

The glow-to-arc transition is known to be triggered by very high current densities impinging on the target or high plasma densities. Both are present in the new HyPIM discharge. The early stage of cathodic spot formation, observed as bright dots, can preserve the glow mode or turn into a hot spot. The energy of cohesion and sublimation of the target material certainly play a key role in the transition to arc [6].

Finally, the co-existence of an arc with a diffusive glow discharge, initiated by a HiPIMS pulse, shows a hybrid glow-arc regime with interesting properties. [7]

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[4] E. Morel, T. Minea, Y. Rozier, Euro. Phys. Lett. (EPL) 138 (2022) 24001; https://doi.org/10.1209/0295-5075/ac2e2b

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2:40pm **PP2-2-WeA-3 Plasma Characteristics, Microstructure, and Mechanical Properties of Tetrahedral Amorphous Carbon Thin Films Deposited by Time-Resolved High-Power Impulse Magnetron Sputtering with Synchronized Bias Control**, ***Fu-Sen Yang* [D11103004@mail.ntust.edu.tw]**, *Yu-Lin Kuo,* National Taiwan University of Science and Technology, Taiwan*; Chi-Lung Chang,* Ming Chi University of Technology, Taiwan, Republic of China

Time-resolved ionization analysis of graphite plasma discharges was conducted using optical emission spectroscopy (OES) and plasma mass spectrometry (PSM) during high-power impulse magnetron sputtering (HiPIMS). During the pulse-on period, the generation sequence of argon and carbon ions is synchronized with the HiPIMS target power supply through the bias control system. The timing of the bias output is then regulated to adjust the incident flux and kinetic energy of these ions, thereby enabling the deposition of a tetrahedral amorphous carbon (ta-C) thin film. The effects on plasma characteristics, microstructure, chemical composition, and mechanical properties were studied. Plasma characteristics were analyzed using time-resolved OES and PSM on a graphite target.Time-resolved analysis revealed that argon ions were generated first, followed by carbon ions. By setting synchronization (Syn.) and delay times (TD = 0, 25, 50, 75, 100, 125, and 150 µs) at the bias trigger, the attraction and arrival sequence of argon and carbon ions at the substrate were controlled, thereby regulating the incident ion flux and energy to facilitate the deposition of the tetrahedral amorphous carbon (ta-C) thin film. The thin film analysis results indicate that all ta-C thin films deposited under different trigger synchronization and delay times exhibit an amorphous structure. However, transmission electron microscopy (TEM) analysis reveals that the crystallinity of carbon nanocrystals improves progressively with increasing delay time. The density of the ta-C thin film was determined using X-ray reflectivity (XRR) analysis, and the results showed that the thin film reached a maximum density of 2.95 g/$cm^3$ at a trigger delay time of 125 µs. The chemical and mechanical analyses revealed that in the synchronous mode, the maximum compressive stress reached -5.6 GPa, the $I_D/I_G$ ratio was 0.52 at a trigger delay time of 125 µs, the $sp^3$ content was 70%, the hardness reached 48 GPa, and the Young's modulus was 263 GPa. This is primarily because a longer trigger delay time allows more carbon ions to be attracted for bombardment while reducing argon ion bombardment, thereby preventing the conversion of $sp^3$ to $sp^2$ bonds caused by thermal effects.

3:00pm **PP2-2-WeA-4 Carbon Discharge Dynamics by Pulse Sequencing: Pulse Parameter Control in Multipulse Hipims**, *Ryo Sakamoto,* ***Tetsuhide Shimizu* [simizu-tetuhide@tmu.ac.jp]**, Tokyo Metropolitan University, Japan

Amorphous carbon (a-C) thin films exhibit excellent mechanical properties. However, a-C films formed by sputtering often show reduced density and hardness due to the low ionization rate of carbon species. The High-Power Impulse Magnetron Sputtering (HiPIMS) technique employs high-density plasma generated by applying short, high-power pulses to the target. A major issue in HiPIMS, however, is ion back-attraction, in which ionized sputtered species are drawn back toward the target by the applied voltage. To address this issue, the multipulse HiPIMS approach applies a train of ultra-short pulses to enhance discharge efficiency through pre-ionization and suppress ion back-attraction, thereby promoting the transport of carbon ions toward the substrate. In this study, the effects of pulse parameters, specifically pulse width and pulse interval on HiPIMS carbon discharge were investigated using energy-resolving time-of-flight mass spectrometry (ETOFMS) during multipulse HiPIMS discharges of a graphite target in an argon atmosphere. The discharge conditions included varying the pulse width to 20, 30, and 50 µs, and the pulse interval to 10, 20, and 50 µs, with the number of sequential pulses fixed at five. Under these conditions, ion energy distribution functions (IEDFs) were measured for $Ar^+$, $Ar^{2+}$, $C^+$, and $C^{2+}$ ions in both time-averaged and time-resolved modes. As results, a high-energy tail was observed in the $C^+$ion energy distribution at shorter pulse width ($t_{on}$), while both $C^+$ and $Ar^+$ion fluxes increased with a higher number of pulses at longer $t_{on}$. In contrast, shortening the pulse-off time ($t_{off}$) led to a significant increase in the ion energy of $C^+$ ions. Furthermore, time-resolved measurements revealed that the $C^+$ ion intensity continued to increase with the number of pulses when $t_{off}$ was reduced to 10 µs, indicating enhanced pre-ionization in after-glow plasma by short pulse interval.

3:20pm **PP2-2-WeA-5 Understanding the Impact of Kinetic and Potential Ion Energies on Thin Film Structure Toward Low-Temperature Deposition**, *Dmitry Kalanov, Andre Anders,* ***Yeliz Unutulmazsoy* [yeliz.unutulmazsoy@iom-leipzig.de]**, Leibniz Inst. of Surface Eng. (IOM), Germany **INVITED**

Over recent years, we have investigated how energetic thin film deposition techniques can reduce conventional substrate temperature requirements, focusing on pulsed filtered cathodic arc deposition. Our work investigates the effect of ion potential energy on thin film structure. The main research questions are: How can the influence of ion kinetic energy, ion potential energy, and ion flux on film structure be studied while decoupling these parameters as much as possible? What is the isolated effect of ion potential energy?

Decoupling these effects is challenging because ion kinetic and potential energies are inherently coupled in cathodic arc plasmas. We applied an external magnetic field to preserve multiply charged ions and conducted comparative studies. The results demonstrate that an increased fraction of multiply charged ions enables the formation of crystalline films without conventional substrate heating. Crystalline, dense, and macroparticle-free metallic (V-Al) and ternary nitride (V-Al-N) films were successfully deposited at room temperature, facilitated by the ion potential energy provided by multiply charged ions[1,2], due to a mechanism known as *"atomic-scale heating."*

These insights could help to reduce or partially replace conventional substrate heating in various energetic deposition processes, lowering energy consumption and enabling thin film deposition on temperature-sensitive substrates. This approach can be critical for sustainable surface engineering across various materials systems.

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## Plasma and Vapor Deposition Processes
## Room Town & Country B - Session PP3-ThA

### CVD, ALD, and Laser-based Deposition & Microfabrication Technologies

**Moderators: Carles Corbella**, National Institute of Standards and Technology (NIST)/ University of Maryland, College Park, USA, **Valentina Dinca**, National Institute for Laser, Plasma, and Radiation Physics, Romania, **Frederic Mercier**, CNRS, Grenoble-INP, University Grenoble Alpes, SIMaP laboratory, France, **Takayoshi Tsutsumi**, Center for Low-temperature Plasma Sciences, Nagoya Universit, Japan

1:20pm **PP3-ThA-1 Unveiling the Potential of Transparent Conductive Materials by Atomic/Molecular Layer Deposition: From Process Synthesis to Functionalization*, Abderrahime Sekkat [abderrahime.sekkat@toulouse-inp.fr],*** Univ. Toulouse, CNRS, Toulouse INP, LGC, Toulouse, France., France **INVITED**

From powering renewable energy systems to transforming lighting and data storage technologies, solar cells, electroluminescent displays (ELDs), organic light-emitting diodes (OLEDs), sensors, and printed electronics are driving the next wave of technological innovation. Transparent conductive materials (TCMs) play a key role in enabling and improving the performance of these devices by offering unique advantages for human–device interfaces and information processing. Today, transparent conducting indium tin oxide (ITO) remains the most widely used TCM, thanks to its excellent optical transparency (>90%) and low sheet resistance (<30 Ω/sq)1,2. It currently holds about 55% of the transparent conductive electrode (TCE) market in 20243. However, ITO is brittle, which limits its use in flexible devices, and its dependence on indium, a critical and scarce resource, raises sustainability concerns. To address these limitations, several alternative TCMs are being actively explored, covering inorganic, metallic, and organic material families.In this presentation, I will give an overview of our ongoing work on developing alternative TCMs using different vapor-phase deposition (VPD) methods. I will first focus on the growth of oxide films using atmospheric pressure spatial atomic layer deposition (AP-SALD), an innovative alternative to conventional ALD4. Unlike traditional ALD, AP-SALD relies on the spatial separation of precursors within a 3D manifold head rather than sequential gas injection. This approach enables faster deposition over large areas, making it well suited for scalable manufacturing. I will present some recent results on p-type oxides obtained by this method5–7. I will then show how oxide coatings can be used to improve the stability of transparent electrodes based on silver nanowire networks8,9. Finally, I will discuss the development of conjugated conductive polymers using oxidative VPD, with examples of their integration into real devices10. Overall, this work illustrates a comprehensive approach, from process synthesis to device functionalization, aimed at advancing the next generation of transparent conductive materials.References1. Nanomaterials 14, 2013 (2024).2. APL Mater. 9, 021121 (2021).3. https://www.imarcgroup.com/transparent-conductive-films-market. (Accessed: 24th July 2025)4. Adv. Mater. Technol. 2000657, 1–8 (2020).5. Nat. Commun. 2022 131 13, 1–11 (2022).6. Commun. Mater. 2, 78 (2021).7. J. Mater. Chem. A 9, 15968–15974 (2021).8. Adv. Mater. Technol. 8, 2200563 (2022).9. Adv. Mater. Technol. 8, 2301143 (2023).10. ACS Appl. Polym. Mater. 5, 10205–10216 (2023).* Corresponding author e-mail: abderrahime.sekkat@toulouse-inp.fr

2:00pm **PP3-ThA-3 ALD and Aerosol-Assisted CVD for Surface and Interface Control in Perovskite LED Structures*, Francisco Servando Aguirre Tostado [servando.aguirre@cimav.edu.mx],*** Centro de Investigacion en Materiales Avanzados, Mexico **INVITED**

Interfacial defects remain a significant impediment to the development of high-performance, stable perovskite light-emitting diodes (PeLEDs). This work presents a comprehensive approach to surface and interface control via complementary chemical vapor deposition methodologies. We employ Atomic Layer Deposition (ALD) to fabricate nanoscale, conformal metal oxide layers such as $Al_2O_3$, ZnO, $SnO_2$, and NiO that serve as both efficient charge-transport layers and moisture barriers. The self-limiting nature of ALD ensures perfect coverage on complex nanostructures, mitigating current leakage and shunt paths. On the other hand, we use Aerosol-Assisted Chemical Vapor Deposition (AACVD) to deposit the perovskite emissive layer, where the precursor solution mixture ratio controls the composition and crystalline phase. AACVD overcomes the limitations of spin-coating by enabling the deposition of uniform, pinhole-free films over large areas with precise control of cation stoichiometry, thereby enhancing photoluminescence quantum yield (PLQY) and spectral purity. The talk will detail the optoelectronic and morphological characterization of these interfaces and correlate them with the device performance. Our results, demonstrating PeLEDs with improved external quantum efficiency (EQE) and a lifetime extended by an order of magnitude, underscore the transformative potential of combining ALD and AACVD for large-area and low-cost optoelectronic devices.

2:40pm **PP3-ThA-5 In-Plasma XPS: a New Metrology Tool for Semiconductor Process Development and Control*, Andrei Kolmakov [andrei.kolmakov@nist.gov],*** NIST-Gaithersburg, USA **INVITED**

Modern ambient pressure X-ray photoelectron spectroscopy (AP-XPS), in addition to its real-time sub-monolayer sensitivity, now covers the pressure range typical of standard plasma processing applications, naturally expanding the capabilities of AP-XPS for operando plasma-assisted control. We recently demonstrated that XPS spectra can be successfully collected under plasma conditions, extending the application of XPS to plasma-surface-liquid-vapor interactions [1]. In previous work [2], we highlighted the importance of plasma chamber wall reactions on sample surface chemistry and showed that plasma-XPS can capture plasma chemistry both at the surface and in the gas phase. We recently applied plasma-XPS to industry-relevant and realistic poorly conducting surfaces, where we observed anomalous XPS binding energy shifts due to sample charging during an AC plasma exposure. We propose mechanisms that explain these plasma-induced shifts. Additionally, we noted plasma-induced binding energy shifts and peak splitting when measuring XPS from the plasma gas phase. The latter can be used for local diagnostics of the local plasma environment.

Overall, plasma-XPS metrology is a new emerging tool that offers significant potential for advancing real-time diagnostics of plasma-assisted deposition processes, and immediate mitigation strategies to reduce the damage of wafers, which is a well-known challenge in semiconductor fabrication [3].

References

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3:20pm **PP3-ThA-7 Plasma Dynamics in Femtosecond Laser Burst-Mode Ablation of Metals for Thin-Film Applications*, Asma Banshamlan [asma.banshamlan@univ-st-etienne.fr],*** *Florent Bourquard, Anne-Sophie Loir, Jean Pierre Chatelon, Christophe Donnet, Florence Garrelie, Bossuet Lilian,* Université Jean Monnet Saint-Étienne, CNRS, Institut d'Optique Graduate School, Laboratoire Hubert Curien, UMR 5516, F-42023 Saint-Etienne, France

The use of femtosecond laser PLD (fs-PLD) has originally been shown to generate controlled distributions of nanoparticles rather than smooth films. The ultrashort pulse duration, shorter than the electron–phonon coupling time, drives rapid, non-equilibrium phase transitions from the metallic to the ionized state, resulting in nanoparticle formation upon cooling.

However, recent advances in femtosecond laser burst-mode operation (coupling MHz and/or GHz bursts), where multiple ultrashort pulses are delivered in rapid succession, enable precise control over the temporal energy deposition compared to single-pulse irradiation. In this study, we investigate burst-mode ablation of metallic targets, including vanadium and tungsten, under varying burst parameters, using ICCD imaging resolved on the nanosecond timescale. These measurements reveal that intra-burst pulse spacing strongly modulates plasma density, particle emission, and expansion dynamics.

Our results show that GHz burst-mode irradiation produces larger plasmas with controlled vapor plume characteristics, substantially reducing nanoparticle formation and facilitating smoother thin-film deposition. These results are confirmed by thin-film characterizations using electron microscopy and atomic force microscopy, demonstrating the ability to obtain smooth surfaces. This work provides fundamental insights into energy coupling and material removal in fs burst-mode ablation and highlights its potential for tailored plasma-assisted fabrication and optimized vapor deposition processes, particularly for including metals in multilayers or even for optimizing Atomic Layer Deposition.

Pulsed-Laser Deposition (PLD) has attracted a lot of interest due to its non-contact nature, its versatility, ease of co-deposition using several targets, and the possibility to perform deposition in ultra-high vacuum conditions, limiting contamination of samples. However, droplet formation has strongly limited its applications for metallic thin-film generation, particularly in the case of multilayer formation including metals.

4:00pm **PP3-ThA-9 Ultrathin $SiN_X$ Membrane Stability Under Energy Fluxes from Non-Thermal Plasma Discharges Monitored via Nanocalorimetry***, **Carles Corbella [carles.corbellaroca@nist.gov]**,* National Institute of Standards and Technology (NIST)/ University of Maryland, College Park, USA*; Feng Yi, Andrei Kolmakov,* National Institute of Standards and Technology (NIST), USA

Freestanding ultrathin silicon nitride ($SiN_x$) membranes are widely used as an electron, X-ray, and light transparent windows for environmental spectromicroscopy, separation membranes, and in microelectronics, e.g., as in MEMS devices and nanocalorimeters. However, their stability in the plasma environment requires further studies. Here, suspended 100 nm-thick $SiN_x$ membranes have been wafer-scale fabricated on 15 $mm^2$-silicon frames using lithography. A platinum lithographically defined resistive microsensor of 100 nm thickness is deposited on the backside of the membrane, and it is calibrated for thermometry and calorimetry. This energy flux sensor (nanocalorimeter) has been exposed to cold plasmas in a custom-made research reactor equipped with a remote inductively coupled plasma (ICP) discharge source, Langmuir probe, retarding field energy analyzer, and optical emission spectroscopy (OES) channel. Energy fluxes (ions, electrons, energetic neutrals, and photons) from plasma plume are registered via sensor temperature evolution upon variations in the plasma parameters. The power carried by plasma species can be estimated from a simple energy balance model in measurements using sensor temperature variations up to a few hundred Kelvin with time resolution below 40 ms [Diulus et al, J. Vac. Sci. Technol. B 43, 020601 (2025)]. Additionally, the measurement setup allows for decoupling of the heating contributions by ions and VUV/UV-Vis-IR photons. It was found that the lifetime of the sensor is defined by the $SiN_x$ sputtering rate combined with thermally induced mechanical stress. Ultrathin $SiN_x$ membranes appear to be very robust even when immersed in the RF plasma plume region, manifesting low sputtering yield under typical electrically grounded experimental conditions. To investigate the chemical stability of the ultrathin membranes, nanocalorimetry experiments in argon plasma have been followed by preliminary tests using reactive gases such as oxygen and hydrogen.

4:20pm **PP3-ThA-10 Conformal PEALD-SiNx Passivation for GaN Vertical SBDs: A Microfabrication Solution to Mesa-Induced Interface Damage***, Jiayu Zeng, **Xuetong Zhou [zhouxuetong@mail.sim.ac.cn]**, Li Zheng, Xinhong Cheng,* State Key Laboratory of Materials for Integrated Circuits, Shanghai Institute of Microsystem and Information Technology, Chinese Academy of Sciences, China

Gallium Nitride (GaN) vertical power devices are pivotal for next-generation high-voltage applications. A critical challenge in their fabrication, particularly for devices utilizing deep mesa etch termination (DEMT) for field management, is the severe surface damage and trench-like defects induced by aggressive etching processes. These defects become dominant leakage paths, severely degrading breakdown voltage and preventing the realization of ideal avalanche breakdown. Conventional dielectric deposition methods like plasma-enhanced chemical vapor deposition (PECVD) struggle to conformally cover these high-aspect-ratio features, leading to interfacial voids and inadequate passivation.

This work presents a microfabrication strategy to overcome this limitation by employing plasma-enhanced atomic layer deposition (PEALD) for conformal silicon nitride ($SiN_x$) passivation. We demonstrate the growth of high-quality $SiN_x$ films via an optimized PEALD process using a $Si_2Cl_6$ precursor under ultra-high vacuum. The process, stabilized at 450°C and 150 W plasma power, yields films with exceptional uniformity (<±1% thickness variation on a 6-inch wafer), low surface roughness (RMS ~0.3 nm), and near-zero oxygen contamination.

The efficacy of this PEALD-$SiN_x$ film was investigated in GaN vertical Schottky barrier diodes (SBDs) with a 6 μm deep mesa. Cross-sectional HRTEM and EDS analysis confirmed that the PEALD-$SiN_x$ provides a conformal, void-free interface, effectively filling etching-induced trenches and mitigating the underlying damaged GaN layer. In stark contrast, PECVD-$SiN_x$ passivation resulted in a defective interface with significant Si diffusion and incomplete coverage. Electrical characterization revealed a transformative improvement in device performance. SBDs passivated with PECVD-$SiN_x$ alone suffered from severe voltage-dependent leakage, reaching 1 mA at just 80 V without breakdown. Conversely, PEALD-passivated devices exhibited a stable, ultra-low leakage current (~$10^{-11}$ A) until a sharp avalanche breakdown at approximately 400 V, a five-fold increase in breakdown voltage and a $10^4$-$10^5$ reduction in leakage. Inter-anode leakage measurements further confirmed that PEALD passivation completely suppressed the sidewall conduction path, restoring bulk-limited conduction.

In conclusion, this study establishes PEALD-$SiN_x$ as an essential micro-fabrication technology for enabling high-voltage GaN vertical devices. Its superior conformality and interface quality directly address the core challenge of etching-induced damage, unlocking the full voltage-blocking potential of DEMT structures and paving the way for robust, high-performance GaN power electronics.

## Plasma and Vapor Deposition Processes
## Room Palm 1-2 - Session PP4-ThA

## Greybox Models for Wear Prediction

**Moderators: Philipp Immich**, IHI Hauzer Techno Coating B.V., Netherlands, **Ludvik Martinu**, Polytechnique Montréal, Canada

2:00pm **PP4-ThA-3 End-to-End Prediction of Coating Properties and Film Distribution via a Digital-Twin Framework***, **Adam Obrusnik [obrusnik@plasmasolve.com]**, Krystof Mrozek, Kristina Stastna, Petr Zikan,* PlasmaSolve s.r.o., Czechia **INVITED**

Physical vapor deposition (PVD) and plasma-enhanced chemical vapor deposition (PECVD) processes generate vast streams of sensor data, waveforms, and post-coat metrology, yet only a small portion of this information informs engineering decisions. This work presents PlasmaSolve's **hybrid digital-twin framework**, a *greybox* approach that combines physics-based plasma and transport simulations with machine-learning models trained on process logs, diagnostic data, and coating metrology. The framework is implemented within the MatSight simulation ecosystem.

The framework enables **end-to-end prediction of key coating properties**, including **film composition, thickness, and their spatial gradients**, as well as **hardness, roughness, or residual stress** - all derived directly from the coating recipe and coating tool layout. These quantities are resolved over complex 3D substrate geometries using the MatSight 3D Uniformity App to produce full-field maps of **mass and energy flux distributions**, linking plasma behavior to the resulting film structure and uniformity.

While the presented models do not directly predict wear progression, they capture the **precursor variables that govern wear performance**, such as local variations in stoichiometry, microstructure, and residual stress. This capability bridges a critical gap between process conditions and the mechanical stability of coatings under service environments. Case studies demonstrate how the hybrid digital twin reduces experimental trial runs by up to **90 %**, accelerates recipe transfer between coating tools, and improves the reproducibility of wear-relevant coating metrics.

The talk outlines the architecture of the greybox framework, emphasizing how simulation-derived features enhance extrapolation beyond the training domain. It concludes with perspectives on extending this approach toward **direct wear and lifetime prediction**, forming a foundation for data-driven process optimization and reliability assurance in advanced thin-film manufacturing.

2:40pm **PP4-ThA-5 Influence of Temperature- Dependent Mechanical Properties on Tool Load in Cutting***, Christian Kalscheuer, Kirsten Bobzin, **Xiaoyang Liu [liu@iot.rwth-aachen.de]**,* Surface Engineering Institute - RWTH Aachen University, Germany*; Benjamin Bergmann, Berend Denkena, Nico Junge,* Institute of Production Engineering and Machine Tools, Hannover, Germany

Hard physical vapor deposition (PVD) coatings are widely applied to protect cutting tools against wear. Simulating the thermomechanical load of coated tools is an important approach to understand wear mechanisms. In previous studies, the PVD coating in finite element chip formation simulations has typically been treated as a rigid body, or its properties were assumed to remain constant in the simulation. However, the mechanical properties of PVD coatings vary with temperature during cutting. Assuming constant properties may therefore reduce simulation accuracy. In this study, the temperature-dependent mechanical properties of a TiAlCrN coating are determined using high temperature nanoindentation, while thermal diffusivity is measured at different temperatures using the laserflash method. These experimentally determined coating properties were integrated in the simulation for the coating. Based on the experimental results the thermomechanical load is then simulated for cutting of C45 steel in a finite element chip formation simulation. The study compares the results of temperature-dependent mechanical coating properties with constant properties. The results show that simulations with temperature-dependent coating properties are different to simulations with fixed coating properties. This represents an advance in the research direction of understanding the thermomechanical tool load during cutting.

3:00pm **PP4-ThA-6 Discovering Hard, Conductive Films via Combinatorial High-Throughput Multimodal Characterization and Machine Learning***,* ***Brad Boyce [blboyce@sandia.gov]****,* Sandia National Laboratories, USA **INVITED**

Hard, electrically conductive films with low friction and high wear resistance are relevant to electrical contact applications. Here we augment traditional process-structure-property investigations with an accelerated workflow to detect material structure/composition, prognose associated properties, and adapt the associated process to achieve improved product outcomes.This accelerated detect-prognose-adapt cycle is aided by four key elements: (1) automated combinatorial synthesis to enable rapid parameter sweeps, (2) high-throughput evaluation of both conventional and surrogate indicators of material chemistry, structure, and properties, (3) machine learning algorithms to unravel correlations in high-dimensional spaces beyond expert cognition, and (4) batchwise Bayesian optimization strategies to balance efficient exploration and exploitation.Unlike other ML-driven materials exploration campaigns that focus on variations in the composition of the material, here our primary emphasis is on variations in deposition conditions.We identify particular deposition conditions that produce metallic thin films with exceptional hardness (>9 GPa), low friction (m<0.1), and low electrical resistivity on par with commercial electrical contact alloys. SNL is managed and operated by NTESS under DOE NNSA contract DE-NA0003525.

## Plasma and Vapor Deposition Processes
## Room Golden State Ballroom - Session PP-ThP
## Plasma and Vapor Deposition Processes Poster Session

**PP-ThP-1 Contact Resistance Improvement of IGZO Devices through the Introduction of a Boosting Technique in the Source/Drain Regions***, **Hyeonmin Jo [hyunmine.cho@g.skku.edu]**, Sungsoo Lee, Jinhong Park,* Sungkyunkwan University (SKKU), Republic of Korea

As silicon-based devices continue to scale down, leakage current has emerged as a critical issue. To overcome this challenge, indium–gallium–zinc oxide (IGZO)–based oxide semiconductors, with their wide bandgap and superior leakage suppression, have been highlighted as promising next-generation channel materials. However, IGZO films face important limitations. Their amorphous structure, relatively low carrier concentration, and high Schottky barrier height (SBH) result in poor contact resistance, which has become a major obstacle for memory applications.

In this study, we propose a novel contact engineering approach that has not been reported previously. Specifically, Carrier boosting has previously been applied to the channel region to modulate the threshold voltage (Vth) of devices. We extend the technique to the source and drain regions. **A boosting layer is selectively deposited using PVD method on the contact areas to enhance doping and improve interface properties**. By locally increasing the doping concentration through this approach, the contact resistance can be effectively reduced. Finally, a high-performance metal electrode is deposited to ensure excellent electrical contact.

This integrated strategy is designed to reduce contact resistance while simultaneously improving the overall device performance.

**PP-ThP-2 CVD-Grown $MoS_2$ Structures: Spiral Growth Mechanisms and Mechanical Response***, **Daniela Lucio-Rosales [daniela.lucio@cimav.edu.mx]**, David Torres-Torres, Alejandra Garcia-Garcia,* Centro de Investigación en Materiales Avanzados S.C., Mexico

Two-dimensional transition metal dichalcogenides (TMDs), particularly molybdenum disulfide ($MoS_2$), exhibit remarkable mechanical and electronic characteristics that make them promising for flexible and strain-engineered devices. However, establishing a direct connection between their growth mechanisms and mechanical performance remains a key challenge. In this work, $MoS_2$ thin films were synthesized by Chemical Vapor Deposition (CVD) on $Si/SiO_2$ substrates, revealing triangular domains dominated by a spiral growth pattern. This morphology originates from screw dislocations that promote a continuous step-flow mechanism during layer formation. The nucleation and propagation of these dislocations are strongly affected by the substrate's surface topography, leading to distinct variations in domain size and layer stacking.

Raman spectroscopy confirmed the crystalline nature of the films, showing well-defined vibrational modes associated with $MoS_2$. The in-plane ($E^1_{2g}$) and out-of-plane ($A_{1g}$) modes appeared around 383 $cm^{-1}$ and 408 $cm^{-1}$, respectively, while additional features near 176 $cm^{-1}$, 228 $cm^{-1}$, and higher-order peaks above 418 $cm^{-1}$ indicated complex interlayer interactions and multilayer coupling. These spectral variations were used to assess the structural uniformity and thickness of the films.

Atomic Force Microscopy (AFM) analyses revealed the stepwise topology typical of spiral growth and enabled quantitative evaluation of the vertical layer spacing. Nanoindentation experiments demonstrated that the presence of screw dislocations enhances the mechanical resilience of the material, resulting in a combined elastic–elastoplastic response. The observed hardening behavior suggests that dislocation-mediated growth can be harnessed to modulate the toughness and strain accommodation of 2D materials.

Overall, this study provides fundamental insight into how growth dynamics influence the microstructural and mechanical characteristics of CVD-grown $MoS_2$. These results contribute to the development of robust and flexible TMD-based coatings for applications in nanoelectromechanical systems (NEMS), strain sensors, and energy-dissipative devices.

**PP-ThP-3 Predictive Modelling of Magnetron Sputtering: Bridging Computational and Experimental Approaches for Metallic Glass Thin Films***, Jaroslav Zenisek, Tereza Schmidtova,* Masaryk University, Czechia*; Antonin Kubicek, Vjaceslav Sochora,* SHM, Czechia*; **Pavel Soucek [soucek@physics.muni.cz]**,* Masaryk University, Czechia

Computational simulations are rapidly transforming the way magnetron sputtering processes are designed, understood, and optimized. They offer a powerful means of increasing experimental efficiency, accelerating process development, and improving reproducibility—particularly when transitioning from laboratory-scale research to industrial-scale coating production. Despite major advances in plasma-assisted deposition, one fundamental challenge remains: precise control of particle sputtering from the target and their subsequent transport toward the substrate. These parameters govern the particle abundance and energy while arriving at the substrate, ultimately determining coating stoichiometry, phase structure, microstructure, and performance.

While compositional gradients and local variations can be highly beneficial for combinatorial thin-film research, they are detrimental in industrial environments, where uniformity in thickness, composition, and phase structure are essential for high-throughput and large-area coating.

In this contribution, we demonstrate the combined use of SDTrimSP and SiMTra simulation tools to predict industrial magnetron sputtering of metallic glasses based on Zr–Cu(–Ni/Al) systems and on W–Ni–B and W–Zr–B systems, representing examples with comparable and strongly contrasting atomic masses. The simulations provide detailed predictions of relative thickness profiles, elemental composition distributions, and the energy spectra of the arriving species under varying process conditions.

The calculated results are compared with experimental data obtained from thin films deposited under controlled conditions, enabling a quantitative assessment of model accuracy and applicability. Furthermore, the functional properties of the deposited metallic glass coatings are correlated with the predicted parameters, establishing a clear link between process simulation and coating performance. This integrated computational–experimental approach provides a valuable framework for scaling magnetron sputtering from laboratory research to robust industrial production of chemically relatively complex coatings.

**PP-ThP-4 How to Predict the Deposition Rate During Reactive Sputtering Using an One-Volume Reference Resource?***, **Diederik Depla [Diederik.Depla@ugent.be]**,* Ghent University, Belgium

A longstanding challenge in reactive magnetron sputtering is the quantitative prediction of the deposition rate, which is primarily determined by the partial metal sputtering yield from the oxide layer formed on the target surface during poisoning. The first step in addressing this issue is to determine the total sputtering yield of the oxide. This has been accomplished by refining a published semi-empirical model. This model has been applied to fit an extensive set of oxide sputtering yield data from the literature, comprising 65 datasets for 21 different materials. The fitting process establishes a relationship between the surface binding energies of metal and oxygen atoms and the cohesive energy of the oxide. The calculated partial sputtering yield of metal from a poisoned target is then compared with previously published experimental data on the metal sputtering yield during reactive magnetron sputtering. While both yields are linearly correlated, the magnetron-based sputtering yields are approximately eight times lower than the model predictions. This reduction in yield is attributed to the formation of an oxygen-rich surface layer, a hypothesis supported by binary collision approximation Monte Carlo simulations. However, these simulations do not fully capture the mechanism, as a more detailed description of the surface oxygen origin is needed. Despite this limitation, the experimental correlation provides a practical strategy for predicting deposition rates during reactive magnetron sputtering in fully poisoned mode. As demonstrated, the oxide sputtering yield can be calculated using standard data sources, and the empirical correlation between the sputtering yields enables a reliable estimate of the metal partial sputtering yield in poisoned mode, thus allowing for an accurate estimation of the deposition rate.

D. Depla, Note on the low deposition rate during reactive magnetron sputtering, Vacuum 228 (2024) 113546D. Depla, J. Van Bever, Calculation of oxide sputter yields Vacuum 222 (2024) 112994

**PP-ThP-5 Properties and Behavior of Infrared Materials : Towards High Efficiency and High Durability Antireflection Coating**, ***Manon Dewynter* *[manon.dewynter@orange.fr]***, *Fabien Paumier, Éric Le-Bourhis, Cyril Dupeyrat,* Institut Pprime - CNRS - ENSMA - Université de Poitiers, France

This PhD research focuses on developing advanced thin-film coatings for substrates with complex geometries, aiming to achieve uniform properties and enhanced resistance under demanding operational environments. The study emphasizes optimizing deposition parameters to ensure consistent film characteristics—critical for the performance and durability of optical components in optronic systems. These systems incorporate diverse optical elements, including windows, lenses, filters, and dichroic plates, all requiring precise functionalization through thin-film treatments to meet stringent optical, mechanical, and chemical specifications.

Front optics in optronic devices play a key role in detection, observation, and identification. Operating under harsh and variable conditions—such as corrosive, erosive, and chemically aggressive environments—these components require coatings that maintain high optical transmission while exhibiting robust mechanical and chemical stability.

Typically, coatings are deposited by vapor-phase techniques such as Electron Beam Physical Vapor Deposition (EB-PVD) and Ion Beam Assisted Deposition (IBAD), enabling the formation of dense, uniform multilayer interference stacks. However, substrates with complex geometries—characterized by large diameters or high curvatures—pose significant challenges for achieving uniform coating deposition. Variations in thickness, density, and mechanical properties across the surface can lead to performance degradation, including chemical attack, delamination, and loss of optical quality, particularly under critical conditions such as saline fog exposure.

This project aims to elucidate the underlying growth mechanisms and physical phenomena at the material scale, focusing initially on single-layer coatings to establish a solid foundation of knowledge. Comprehensive characterizations—including nanoindentation, ellipsometry, and strain measurements—are employed to assess mechanical and optical properties and to study the influence of deposition parameters. The insights gained will guide the design of novel multilayer architectures incorporating new materials and interfaces to enhance thermomechanical performance.

Ultimately, this research supports the evolving specifications of optronic devices by delivering coatings with improved robustness and consistent functional properties, thereby advancing the performance and reliability of front optics in demanding operational environments.

**PP-ThP-6 Plasma Research Reactor to Validate Nanocalorimetry as a Prospective Plasma Diagnostics Technique**, ***Carles Corbella [carles.corbellaroca@nist.gov]***, National Institute of Standards and Technology (NIST)/ University of Maryland, College Park, USA*; Feng Yi, Andrei Kolmakov,* National Institute of Standards and Technology (NIST), USA

Recent advances in microelectronics require techniques for faster, more accurate, and comprehensive characterization of plasma-based nanofabrication processes, such as film deposition and surface etching or cleaning. Our recent demonstration of using membrane-based differential nanocalorimetry to measure atomic radicals in reactive plasmas sensitively [Diulus et al, J. Vac. Sci. Technol. B 43, 020601 (2025)] has inspired the further development of this new plasma probe. This probe aims to analyze plasma parameters and fundamental plasma-surface interactions through heat exchange measurements.The present work describes a research plasma reactor equipped with adjustable ICP and CCP sources and standard plasma diagnostics tools to benchmark the nanocalorimeter output: (1) single and double Langmuir probes to provide plasma parameters and electron energy probability function (EEPF); (2) retarding field energy analyzer (RFEA) with a built-in quartz microbalance to evaluate ion energy distributions and mass variation rates, and (3) optical emission spectroscopy (OES) together with (4) quadrupole mass spectrometer for plasma/wall chemistry monitoring. Key nanocalorimeter characteristics, such as sensitivity, response time, lifetime, and stability, as well as parasitic signal interference, will be discussed. This new sensor is well-suited for monitoring surface modification processes in multiple plasma treatment applications.

**PP-ThP-7 The Role of ALD Oxide Interlayer Thickness in Metal-Polymer Adhesion**, ***Johanna Byloff [johanna.byloff@empa.ch]***, ETH Zurich, Switzerland*; Claus Trost,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben, Austria*; Vivek Devulapalli, Barbara Putz,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland

Adhesion of metal-polymer thin film systems remains a critical challenge for flexible electronics, where interfacial failure can compromise device performance [1]. Here, we demonstrate how atomic layer deposition (ALD) of ultra-thin $AlO_xH_y$ interlayers systematically improves the interface quality of aluminum films on polyimide substrates. Using a novel ALD-PVD cluster system with in-vacuo transfer for precise control of interlayer thickness from 0.14 to 25 nm, we identify four distinct interfacial regimes. Distinction is based on structural and chemical transmission electron microscopy analysis of the interfacial region correlated with delamination, crack initiation and propagation during uniaxial tensile testing studied with in situ electrical resistance measurements and post-mortem atomic force microscopy. Ultra-thin ALD coverage (0.14 nm) enhances native Al-O-C complex formation and adhesion, while a 1 nm-thick ALD later results in unfavorable island-growth coverage, which generates an inhomogeneous interface acting as stress concentrators. At optimum intermediate thicknesses (5-10 nm), a homogeneous and ductile $AlO_xH_y$ interface is formed, while thicker interlayers (≥ 25 nm) induce embrittlement following an intrinsic ductile-to-brittle transition. We show that a 5-10 nm ALD interlayer facilitates plasticity in the Al layer, which results in a 35% increase in interfacial shear strength and maintained electrical conductivity up to 8% applied strain. These findings provide a framework for engineering robust metal-polymer interfaces without the embrittlement associated with traditional adhesion-promoting interlayers, offering pathways for enhanced reliability in flexible electronics applications.

[1] Lu M., You J., Gao M., Li W., Zhang C., Zhu B., Peng C., Wu S., Ren W., Li G., Guo C. F., Yang J., Interfacial adhesion in flexible electronics: Materials, structures and applications, Coordination Chemistry Reviews, Volume 523 (1), 2025, 216278

**PP-ThP-8 Ion Acceleration on Insulating Substrates: Synchronized Floating Potential HiPIMS for AlN and AlScN Thin Film Growth**, ***Oleksandr Pshyk [oleksandr.pshyk@empa.ch]***, *Jyotish Patidar, Kerstin Thorwarth, Lars Sommerhäuser, Sebastain Siol,* Empa - Swiss Federal Laboratories for Materials Science and Technology, Switzerland

Ion acceleration is one of the main process tools in the field of ionized physical vapor deposition (IPVD) for thin-film microstructure manipulation. However, the acceleration of film-forming ions onto insulating substrates has been limited, if not impossible, using conventional approaches. Recently, the demonstration of synchronized floating potential HiPIMS (SFP-HiPIMS) has opened new avenues for controlled metal-ion acceleration on insulating substrates [1].

In this presentation, we report on systematic studies of two industrially relevant materials – AlN and AlScN thin films – grown on a range of insulating substrates using SFP-HiPIMS. The substrates include single-crystalline silicon (001), Z-cut quartz, c-cut sapphire, and amorphous borosilicate glass. The concept of SFP-HiPIMS is based on synchronizing the arrival of film-forming ions at the substrate surface with the build-up of the negative floating potential. Since the sputtered species in HiPIMS are time-separated and the build-up of the negative floating potential is transient, achieving this requires precise synchronization between the HiPIMS pulse-on time and the floating potential-on time (defined as the time offset). Such synchronization allows the attraction of film-forming ions to the substrate while avoiding $Ar^+$ process gas ion bombardment and incorporation into the growing film. Although the SFP-HiPIMS can be implemented using at least two HiPIMS pulses, we demonstrate its feasibility not only for a multiple-magnetron configuration but also for a single-magnetron setup. We evaluate the microstructural quality of AlN and AlScN thin films grown by conventional HiPIMS and SFP-HiPIMS under different magnetron configurations and time offsets in terms of rocking-curve full-width at a half maximum (FWHM), Ar content, and surface roughness.

References:

[1] Nature Communications (2025) 16:4719

**PP-ThP-9 Effect of Si and B Incorporation in TiCN-based Thin Film on Physical Properties by Direct Current Plasma Chemical Vapor Deposition***,* ***Rizu Kurogi [ss25435t@st.omu.ac.jp]****, Takeyasu Saito, Noki Okamoto, Mika Kawamoto,* Osaka Metropolitan University, Japan

Ti-based carbonitride thin films such as TiN, TiC, and TiCN have been used to enhance wear resistance and lifetime of cutting tool. Recent studies employed additional elements such as Si or B to form multi component thin films like TiSiCN and TiBCN to improve oxidation resistance and thermal stability. These films are considered to consist of Ti(C,N) nanocrystals dispersed in amorphous TSiCN or TiBCN, which effectively suppresses grain coarsening and also enhances oxidation resistance.

However, most of the TiSiCN and TiBCN thin films have been synthesized to date by physical vapor deposition methods such as magnetron sputtering or arc evaporation, which often result in poor step coverage and interfacial adhesion strength on complex-shaped substrates. Most of previous studies employed physical vapor deposition methods such as magnetron sputtering or arc evaporation, while plasma enhanced chemical vapor deposition (CVD) provides potential advantage on higher conformality, stronger interfacial adhesion and low temperature fabrication for complex-shaped tools and components.

In this study, TiSiCN and TiBCN thin films were deposited on Si and cemented carbide (WC–Co) substrates using direct current plasma CVD at around 600 ℃ where WC–Co substrates were pretreated with aqua regia to improve interfacial adhesion.

The precursor gases were $TiCl_4$, $CH_4$, $N_2$, tetramethyl silane (TMS), and $BBr_3$. The effects of deposition parameters on the film structure and physical properties were systematically investigated using X-ray diffraction, X-ray photoelectron spectroscopy, and nanoindentation.

Si content in the TiSiCN thin films increased with increasing TMS flow rate, while the B content in the TiBCN thin films also increased with increasing $BBr_3$ flow rate. TiSiCN thin films exhibited higher hardness as maximum value of HV 2585 than that of TiCN thin film. However, the hardness of TiSiCN film decreased according to increase of Si content in the film. The effects of addition of Si and B on grain refinement and structural densification will be discussed.

**PP-ThP-10 RF Plasma Nitriding of Quartz***,* ***Stephen Muhl [muhl@unam.mx]****, Julio Cruz,* UNAM, Mexico*; Enrique Camps,* ININ, Mexico

Plasma nitriding is a valuable and well-established technique for surface hardening of metals to improve their mechanical and tribological properties, such as hardness and wear resistance. Typically, plasma nitriding involves the use of a glow discharge of a mixture of nitrogen and hydrogen, where the metal component to be treated is the cathode and the chamber wall is the anode. The low-pressure plasma (15–1500 Pa) produced by the application of a DC potential (0.3–1.0 kV) contains nitrogen ions, which are accelerated towards the cathode and implanted in the surface of the metal. The treatment time, surface concentration of nitrogen, and temperature of the metal component determine the depth and gradient of the nitride layer, but various tens of microns are often formed. The same process cannot directly be used to nitride insulators since such materials cannot be used as an electrode in a DC plasma.

We have developed a variant of the normal plasma nitriding scheme where the discharge is produced by applying an RF potential and the piece to be nitrided is mounted on a magnetron cathode. This is, of course, the same as a RF magnetron sputtering system. It is known that the DC voltage bias generated on the surface of such an insulting target with a RF magnetron sputtering cathode depends on the following factors: the relative areas of the anode and cathode, the applied RF power, the gas pressure and composition, and the degree of matching of the impedance of the electrical supply to the impedance of the plasma. We have measured the DC bias potential and the rate of sputtering of a quartz target mounted on a 2” MAK magnetron cathode in a pure nitrogen gas discharge as a function of the area of the anode, the applied power, and the degree of matching indicated by the ratio RF(Reflected Power) / RF(Forward Power). Using conditions which produced a minimum sputter etching of the target, we produced three nitrided samples. We present the dimensions and composition of the nitride layers measured using XPS and RBS, the hardness and wear resistance and the optical properties measured using multi-wavelength ellipsometry of these layers.

## Plasma and Vapor Deposition Processes
## Room Palm 5-6 - Session PP2-3-FrM

### HiPIMS, Pulsed Plasmas, and Energetic Deposition III

**Moderators: Arutiun P. Ehiasarian**, Sheffield Hallam University, UK, **Tetsushide Shimizu**, Tokyo Metropolitan University, Japan

8:00am **PP2-3-FrM-1 Experiments and Modelling of High Power Impulse Magnetron Sputtering Discharges with Metallic Target***, **Jon Tomas Gudmundsson [tumi@hi.is]**, Kateryna Barynova,* University of Iceland*; Martin Rudolph,* Leibniz Institute of Surface Engineering (IOM), Germany*; Joel Fischer,* Linkoping University, Sweden*; Tetsuhide Shimizu,* Tokyo Metropolitan University, Japan*; Daniel Lundin,* Linkoping University, Sweden

High power impulse magnetron sputtering (HiPIMS) discharges with a number of metal targets have been explored experimentally followed by a further study using the ionization region model (IRM). The metal targets studied include, tungsten [1], chromium [2], zirconium [3], titanium [4], and copper [5]. Experimentally, the ionized flux fraction has been found to be in the range 10 - 80 %, and it is found to increase with increased discharge current density, and decreased working gas pressure. However, the deposition rate generally decreases with increased peak discharge current density. There is a trade off between high ionized flux fraction and high deposition rate, sometimes referred to as the HiPIMS compromise. An overview will be given on the experimental results for various target materials and dependence on varying operating parameters such as peak discharge current density and pulse length. The IRM allows for studying the temporal evolution of the discharge current composition, the electron power absorption mechanisms, the ionization and back-attraction probabilities of the sputtered species, the dominant recycling mechanism, and the working gas rarefaction. We discuss how the discharge current composition varies between different target materials, and how the recycled species, and the processes leading to working gas rarefaction, depend on the target sputter yield [4]. In particular we will discuss how the back-attraction probability of the sputtered species depends on the sputter yield of the target material [7].

[1] Swetha Suresh Babu et al., Plasma Sources Science and Technology, 31(6) (2022) 065009

[2] K. Barynova et al. Plasma Sources Science and Technology, submitted 2025

[3] Swetha Suresh Babu et al., Journal of Vacuum Science and Technology A, 42(4) (2024) 043007

[4] T. Shimizu et al. Plasma Sources Science and Technology, 30(4) (2021) 045006

[5] J. Fischer et al., Plasma Sources Science and Technology, 32(12) (2023) 125006

[6] K. Barynova et al., Plasma Sources Science and Technology, 33(6) (2024) 065010

[7] K. Barynova et al., Plasma Sources Science and Technology, 34(6) (2025) 06LT01

8:20am **PP2-3-FrM-2 Knowing and Controlling the Dynamic Plasma Potential and Sheath Voltage as Key Elements in Plasma-Based Deposition***, **André Anders [andre.anders@plasmaengineering.com]**,* Plasma Engineering LLC, USA **INVITED**

It is widely known that a space charge layer exists between plasma and a surface (target, substrate, wall, probe, etc.) which is called the sheath.The sheath voltage is the difference between the surface potential and the potential at the sheath edge, the boundary between plasma and sheath. Space charge is linked via the Poisson equation to an electric field which governs fluxes of charged fluxes and thereby energy delivered to the surface.There is nothing new so far, but in real life, for practical reasons, one uses (earth) ground as the reference, not the plasma potential.This can lead to confusion, especially as the plasma potential is not constant in space and time when using modern approaches to plasma-based deposition that involves magnetic fields and pulsed processing, such as bipolar HiPIMS.In this contribution, the establishment of plasma potential, or better the dynamic plasma potential distribution, will be explored and the consequences for film growth discussed.The local and dynamic plasma potential can be associated with numerous effects such cathode spot and anode spot formation (a.k.a. “arcing” and “fireball” in magnetron systems, respectively), the control of ion and electron flows, which affect a growing film’s microstructure, and also with unwanted effects such as sputtering of and arcing on chamber walls and other grounded components.Knowing and controlling the dynamic plasma potential and sheath voltage is therefore important to plasma-based deposition processes.

9:00am **PP2-3-FrM-4 Electrocatalytic Performance of AlCrCoNiFeX (X = C, O)HighEntropy Alloy Films for Oxygen and Hydrogen Evolution Reactions***, **Amna Waheed [amnawaheed146@gmail.com]**,* Ming Chi University of Technology, Taiwan*; Bih-Show Lou,* Chang Gung University, Taiwan*; Jyh-Wei Lee,* Ming Chi University of Technology, Taiwan

The growing demand for sustainable and efficient energy conversion technologies has intensified interest in developing advanced electrocatalysts for water splitting. Highentropy alloys (HEAs), composed of multiple principal elements in near-equiatomic ratios, offer a promising platform due to their unique compositional flexibility, tunable electronic structure, and synergistic catalytic effects. In this work, AlCrCoNiFeX (X = carbon and oxygen) HEA films were synthesized via reactive HiPIMS to assess their bifunctional electrocatalytic activity for the oxygen and hydrogen evolution reactions (OER and HER) in alkaline media. The carbon and oxygen contents were systematically varied to study their combined effects on the structural, morphological, and electrochemical properties of the deposited HEA films. The enhanced catalytic behavior can be ascribed to the synergistic interactions among multiple metallic constituents and the optimized surface structure resulting from carbon–oxygen–carboxylic incorporation. Electrochemical evaluations, including linear sweep voltammetry (LSV), electrochemical impedance spectroscopy (EIS), and double-layer capacitance ($C_{dl}$) measurements, confirmed the superior charge transfer kinetics, larger electrochemically active surface area, and improved catalytic efficiency of the optimized composition. Furthermore, long-term stability and durability tests demonstrated excellent sustainability of the catalyst under continuous operation, validating its structural robustness and electrochemical reliability. This study highlights the potential of AlCrCoNiFeXHEA films as a new generation of efficient and durable bifunctional electrocatalysts for practical water-splitting applications.

# Author Index

## Bold page numbers indicate presenter