

## 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 Spot Stabilization and Thin Film Synthesis Using an Industrial-Sized DC Vacuum Arc Source with Magnetic Steering and Zr-Cu/Zr-Ni Cathodes, Igor Zhirkov [igor.zhirkov@liu.se], Andrejs Petruhins, Linköping University, Sweden; Philipp Immich, IHI Hauzer Techno Coating B.V., Netherlands; Szilard Kolozsvari, Peter Polcik, PLANSEE Composite Materials GmbH, Germany; Johanna Rosen, Linköping University, Sweden**

Zirconium-based alloys, due to their wide spectrum of properties, are broadly used in various applications, ranging from nuclear reactors to biomedical devices. These alloys are characterized by a favorable combination of high glass-forming ability, high thermal stability, and excellent mechanical properties. Additionally, they exhibit excellent anticorrosion behavior, making them attractive for applications in machinery, microelectronics, and aerospace. Furthermore, the cost of synthesizing Zr-based alloys is relatively low compared to that of other materials, making them economically feasible for large-scale applications. However, reports on the deposition of Zr-Cu and Zr-Ni coatings using DC vacuum arc are very limited, even though this process is commonly used in industry. In this work, we present an analysis of the stability of the arcing process based on the corresponding phase diagrams and the presence of Zr, Cu (or Ni) material grains within the cathode. We show that arcing can be stabilized by utilizing a magnetic arc steering system. The study was performed using an industrial-scale arc plasma source, the Hauzer CARC+, which utilizes planar cathodes 100 mm in diameter. The  $Zr_{1-x}Cu_x$  and  $Zr_{1-x}Ni_x$  ( $x = 0.05, 0.10$  at.%) cathodes were provided by PLANSEE Composite Materials GmbH. The magnetic arc steering system, based on variation of the electrical current in a solenoid placed behind the cathode, allows tuning of the magnetic field strength at the center of the cathode surface. In this work, using a Hiden EQP mass-energy analyzer, SEM, and XRD, we demonstrate the plasma and resulting film properties over a relatively wide range of operational pressures (from a base pressure of  $1 \times 10^{-5}$  Torr to  $5 \times 10^{-2}$  Torr) in the reactive atmosphere of  $N_2$  used here. At base pressure, plasma analysis shows ion energies consistent with the velocity rule: approximately  $\sim 70$  eV and  $\sim 50$  eV for Zr and Cu, respectively, and  $\sim 65$  eV and  $\sim 40$  eV for Zr and Ni, respectively. In turn, the plasma ion compositions approximately match the compositions of the corresponding cathodes. The plasma properties were correlated with those of the deposited thin films, including their composition and structure across the full range of studied  $N_2$  pressures. In addition, some evaluations of the resulting films were performed using CIELAB color measurements. The results show that DC vacuum arc deposition can be used for Zr-Cu and Zr-Ni layer depositions.

**2:00pm PP1-2-MoA-2 Relationship Between Substrate Bias and Hydrogen Barrier Behavior of Pulsed DC ZrN Thin Films on Zircaloy-4 Deposited by RF Magnetron Sputtering, Cheng-Han Wu [Jordan91618@gmail.com], Kuan-Che Lan, National Tsing Hua University, Taiwan**

The mechanical integrity of Zircaloy-4 claddings used in light-water reactors is closely related to hydrogen-induced degradation during service. To suppress hydrogen ingress, zirconium nitride (ZrN) thin films were deposited on Zircaloy-4 substrates using an RF magnetron sputtering system operated in pulsed DC mode. This study investigates the relationship between substrate bias and the hydrogen barrier behavior of ZrN coatings by systematically varying the substrate bias during deposition.

Surface morphology and hydride layer formation were examined using scanning electron microscopy (SEM) and focused ion beam (FIB) techniques. Phase constitution, crystallographic texture, grain size, and residual stress were characterized by X-ray diffraction (XRD) and grazing-incidence XRD (GIXRD). Compositional characteristics, including the nitrogen-plus-oxygen-to-zirconium ratio and elemental depth distribution, were analyzed using electron probe microanalysis (EPMA) and X-ray photoelectron spectroscopy (XPS).

The relationship between substrate bias, microstructural features, and hydrogen resistance of the ZrN films is discussed, providing insight into process-structure-property correlations for ZrN coatings deposited by RF magnetron sputtering in pulsed DC mode for zirconium-based nuclear cladding applications.

**Keywords:** Zirconium nitride ZrN, thin films, Substrate Bias, Hydrogen Permeation Barrier, Magnetron Sputtering

**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  $\mu\text{m}$  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.

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**2:40pm PP1-2-MoA-4 Structural Transformation and Electrical Transport in Magnetron-Sputtered Pr-Ni-Co Thin Films, Bisheswor Acharya [bish5197@vandals.uidaho.edu], You Qiang, Xavier Naranjo, University of Idaho, USA; Wenjuan Bian, Haixia Li, Idaho National Laboratory, USA; Hanping Ding, The University of Oklahoma, USA; Thomas Williams, University of Idaho, USA**

Understanding and controlling the physics of alloy thin films with designed microstructure and tunable electronic response is essential for enabling emerging nanoscale devices. However, simultaneously achieving structural continuity and efficient charge transport remains difficult. In this work, we deposit Pr-Ni-Co (PNC) alloy thin films via DC magnetron sputtering and reveal how deposition time directs their growth trajectory. At early growth stages, the films consist of isolated metallic domains; with longer deposition, these features rapidly merge into a continuous, well-ordered layer. This change is accompanied by a sharp decrease in electrical resistance, indicating the formation of a fully connected conduction network. These results show that deposition duration is a key lever linking microstructural evolution to electronic transport in rare-earth-transition-metal alloys, highlighting PNC thin films as a versatile platform for next-generation electronic, magnetic, and catalytic applications.

**3:00pm PP1-2-MoA-5 From Anode-Assisted Magnetron Sputtering to Newer Developments Such as Inverted Fireball-Assisted Magnetron Sputtering, Martin Fenker [fenker@fem-online.de], fem Research Institute, Germany**

#### INVITED

Auxiliary anodes have already become an integral part of sputtering technology for some equipment manufacturers: by providing an additional electron sink at positive potential, they reshape current paths, reduce wall losses, and help to "fill" the coating volume with plasma. In multi-cathode arrangements, a positively biased central anode can strongly increase the substrate current and thus enable a permanent, low-energy ion bombardment even on three-dimensional parts and shadowed surfaces during film growth. In related advanced sputter concepts, remote/common

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anodes biased positive to the chamber wall are used to tune gas/metal ion densities in the coating volume and support stable deposition conditions.

Starting from this classical anode-assisted magnetron sputtering concept, this presentation moves to “active” anodes that do more than stabilize the discharge. A transparent, biased grid anode can act as a plasma-forming element and create an inverted fireball (IFB): a dense plasma confined within the grid with a highly homogeneous plasma potential. In an Ar discharge, IFB ignition requires an anode potential above the gas ionization threshold. In a magnetron environment IFB ignition was demonstrated with a minimum anode voltage of about 22–23 V and can be operated at typical sputter pressures.

The final part focuses on IFB-assisted magnetron sputtering results from Ti deposition on high-speed steel. Multipole resonance probe measurements show electron densities around  $2 \times 10^{16} \text{ m}^{-3}$  and electron temperatures of about 4 eV in the IFB. Optical emission spectroscopy confirms the presence of Ti ions, consistent with enhanced ionization in the combined magnetron–IFB plasma. Relative to conventional dcMS and to a simple +100 V anode plate, IFB assistance yields a markedly denser Ti film morphology and a pronounced hardness increase (e.g., 429→523 HV, +22% without substrate bias). Together, these results position IFB-assisted magnetron sputtering as a low-complexity route to higher ion flux and improved film performance without sacrificing continuous-process deposition rates.

4:00pm **PP1-2-MoA-8 Particle, Momentum and Energy Fluxes in PVD Processes - Probe Diagnostics Are Still in Vogue?**, *Holger Kersten* [[kersten@physik.uni-kiel.de](mailto:kersten@physik.uni-kiel.de)], Kiel University, Germany **INVITED**

4:40pm **PP1-2-MoA-10 High Fidelity Discrete Element Modelling of Particles in Motion for PVD Coating Optimization**, *Faranak Tayefi Ardebili* [[faranak.tayefiardebili@unamur.be](mailto:faranak.tayefiardebili@unamur.be)], University of Namur, Namur Institute of Structured Matter (NISM), Namur, Belgium, USA; *Jerome Muller, Pavel Moskovkin, Cedric Vandenaabeele, stephane Lucas*, University of Namur, Namur Institute of Structured Matter (NISM), Namur, Belgium

Achieving efficient thin-film deposition using PVD methods can be particularly challenging, especially when working with complex geometries or multi-component assemblies where shadowing effects may arise. This is evident in applications implying millimetre to micrometer scale particles, such as ball bearing or battery powder, where uniform coverage is critical. But coating uniformity strongly depends on particle motion and is difficult to quantify experimentally.

In this study, we investigate thin films deposited by magnetron sputtering onto small beads placed inside a vibrating container. The aim is to determine whether conformal coatings can be achieved using this approach, and to identify the conditions required to do so. For that, particle dynamics are simulated using the Discrete Element Method (DEM) implemented in the LIGGGHTS open source code with a Hertz Mindlin contact model. Simulated trajectories are post processed to extract velocity fields, circulation regimes, height distributions, and mixing indices. Predictions are compared with high speed video recordings, showing circulation pattern, and mixing time across a range of excitation amplitudes. From these trajectories we construct spatial maps of collision frequency and particle residence time in a plasma active zone as proxies for coating exposure.

To go further, the PVD process itself can be modelled using the Virtual Coater platform, which combines Monte Carlo model and ray-tracing techniques to compute flux distribution across particle surfaces and predict film properties. This framework identifies motion settings that improve exposure uniformity and reduce dead zones, providing a practical tool for process screening and optimization.

5:00pm **PP1-2-MoA-11 Investigation on Surface Properties Evolution during PVD Duplex Coating Production Steps for H13 Hot Work Steel**, *João Vitor Piovesan Dalla Nora*, Federal University of Rio Grande do Sul, Brazil; *Felipe Canal*, Universidade Federal do Rio Grande do Sul, Brazil; **Leandro Bettoni Ortega** [[leandro.bettoni.ortega@gmail.com](mailto:leandro.bettoni.ortega@gmail.com)], Oerlikon Balzers, USA; *Steffen Aichholz, Rafael Lopes da Silva*, Oerlikon Balzers, Brazil; *Alexandre Da Silva Rocha*, Universidade Federal do Rio Grande do Sul, Brazil  
AISI H13 tool steel is widely used in hot forging applications, where service life is predominantly limited by surface-related failures such as abrasive wear and plastic deformation. While duplex treatments combining plasma nitriding and Physical Vapor Deposition (PVD) coatings have demonstrated significant improvements in surface hardness, wear resistance, and load-bearing capacity, a systematic understanding of how surface integrity changes throughout the entire multi-stage manufacturing sequence - from

heat-treated substrate to finished product - remains lacking. This study addresses this gap by characterizing the evolution of surface properties across sequential processing modifications. Adhesion assessment indicated satisfactory coating-substrate bonding, attributed to the synergistic effects of the nitrided load-supporting layer and microblasting-enhanced mechanical interlocking. Wettability measurements showed a hydrophobic final surface. Following hot forging cycles, quenched and tempered dies exhibited more aggressive wear and geometry loss than duplex-treated dies. Failures and cracks on the latter were attributed to substrate hardness loss. The findings provide a holistic framework for optimizing duplex treatment parameters, particularly the integration of microblasting as both intermediate and final steps, to enhance surface quality, coating adhesion, and potential service performance of AISI H13 dies. Stages applied to quenched and tempered AISI H13: high-speed milling, mechanical polishing, low-pressure gas nitriding, intermediate microblasting, plasma nitriding, and PVD coating (CrAlTi-based), and final microblasting. At each stage, surface integrity was assessed through roughness, three-dimensional topography parameters, surface and cross-sectional morphology, coating adhesion, and wettability. The performance of the duplex-treated dies was further evaluated against the quenched and tempered baseline through laboratory-scale hot forging cycles under critical conditions. Results reveal that each treatment step imparts distinct modifications to the surface. Gas nitriding produced a compound-layer free diffusion zone, increasing subsurface hardness. Intermediate microblasting significantly altered surface morphology, increasing roughness and promoting mechanical anchoring sites for subsequent coating deposition. The PVD process applied via cathodic arc introduced characteristic droplets and porosity, increasing surface roughness. Final microblasting at reduced pressure attenuated these defects, reducing the presence of droplets and peak heights, as inferred from topography.

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