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

## Plasma and Vapor Deposition Processes Poster Session

**PP-ThP-2 Influence of the Substrate on the Growth of Aluminium Oxide Films by Atomic Layer Deposition for Food Packaging Applications**, *Hugo Patureau,* SIMaP, CNRS, University Grenoble Alpes, France*; Thierry Encinas,* CMTC, Grenoble INP, University Grenoble Alpes, France*; Alexandre Crisci,* ***Frederic Mercier [frederic.mercier@grenoble-inp.fr]***, SIMaP, CNRS, University Grenoble Alpes, France*; Erwan Gicquel,* CILKOA, France*; Arnaud Mantoux, Elisabeth Blanquet,* SIMAP, CNRS, University Grenoble Alpes, France

With the gradual ban on single use plastics, cellulosic products have emerged as suitable candidates to replace plastics in the packaging industry. Cellulose is biodegradable, recyclable and possesses good mechanical properties. To be viable for packaging, especially in the food industry, cellulose surfaces need to be functionalised to obtain additional properties, such as wettability, oxygen/water barriers and mechanical resistance in humid conditions.

In this context, we have investigated the synthesis of aluminium oxide films by an industrial Atomic Layer Deposition (ALD) process on cellulosic substrates using the precursors trimethylaluminium (TMA) and water. While the reactivity of these precursors on silicon are well established, the same cannot be said of cellulosic substrates due to their complex structure and their affinity with water. In this presentation, a study on the growth of ALD Al2O3 on silicon and cellulose is conducted. X-ray fluorescence (XRF) and Inductively coupled plasma mass spectrometry (ICP-MS) on cellulose is developed and implemented to quantify the amount of aluminium deposited. The saturation curves are established on silicon and cellulose, as well as the effect of the synthesis temperature. A comparison of both substrates is made and specific growth mechanisms of aluminium oxide by ALD on cellulosic substrates is discussed.

**PP-ThP-3 Minimizing Secondary Electron Yield in Amorphous Carbon Thin Films: A Study on Power Density, Discharge Modes, and Hydrogen Incorporation**, ***Valentine Petit [valentine.petit@cern.ch]***, *Yorick Delaup, Alessia Pascali, Pedro Costa Pinto, Marcel Himmerlich, Christos Kouzios,* European Organization for Nuclear Research, Switzerland

Amorphous carbon thin films with low Secondary Electron Yield (SEY) are critical for applications where electron multipacting limits achievable performance. Such films are effective to mitigate electron cloud formation within the vacuum beam lines of particle accelerators such as the Large Hadron Collider and Super Proton Synchrotron at CERN. They are now also being implemented in the new Electron Ion Collider under construction at Brookhaven National Laboratory.

Research over the last decade has highlighted the significant role of hydrogen presence in the plasma discharge during deposition. Hydrogen incorporation in the films has been shown to increase the SEY, posing a key challenge in coating the extensive beam pipes for particle accelerators.

In this study, we examine the effects of power density and discharge mode, i.e. Direct Current (DC) and High-Power Impulse Magnetron Sputtering (HiPIMS), on the SEY of amorphous carbon films. These films were produced by sputtering in an Ar atmosphere with 1.3% $D_2$ to simulate hydrogen-like impurities typically arising from outgassing in the beam pipes and the deposition system. The $D_2$ consumption during the coating process was monitored by mass spectrometry and is correlated with the SEY, while X-ray Photoelectron Spectroscopy was used to characterize the films. Our findings indicate that higher deposition powers result in films with reduced deuterium incorporation and lower SEY. Additionally, for the same average power density, films deposited in HiPIMS mode exhibit lower SEY compared to those deposited in DC mode. The results are discussed in the context of hydrogen incorporation mechanisms in carbon films, with a view toward optimizing coating system design and process parameters

**PP-ThP-4 Accurate Reporting of Time-of-Flight Measurements with Gated Mass Spectrometry**, ***Nathan Rodkey [nathan.rodkey@empa.ch]***, *Jyotish Patidar, Sebastian Siol,* EMPA (Swiss Federal Laboratories for Materials Science and Technology), Switzerland

The quality of high-power impulse magnetron sputtering (HiPIMS) deposited films can often be improved through the effective use of metal-ion synchronization (MIS). However, effective synchronization requires precise measurements of the time-of-flight (ToF) of ions, such that an accelerating bias can be properly synchronized. These measurements are commonly done using time- and energy- resolved mass spectrometry but require calibrations of the transit time of ions inside of the mass spectrometer to accurately report the ToF. The transit time can be calculated by estimating the travel length in varying parts of the spectrometer (e.g. from orifice to detector) and accounting for the interactions of ions with varying electrostatic optics (such as the extractor, energy filter, mass filter, and dynode).The errors associated with these estimations can lead to nonphysical values in a HiPIMS process, such as negative ToFs, or metal ions arriving to the substrate before process gas ions. As a result, many groups emphasize that their calibrations are estimations, or relevant only at sufficiently large time steps. Here we report a practical approach to determine the transit time in the spectrometer experimentally, which was already successfully employed for multiple projects in our group**.**We use a bipolar HiPIMS power supply to synchronize a gating pulse to the front end of a HiDEN Analytical EQP-300 mass spectrometer. The orifice of the mass-spec (50 µm) was placed at a 12 cm working distance. ToF was then measured by applying a +70 V bias to repel ions, and a 5 µs gating pulse of -30 V to accept them. To prevent interference of the driven front end (kept at +70 V) with the HiPIMS plasma, a grounded shield is placed in front of the mass-spec head with a 1-2 mm opening. The gate was synchronized to the HiPIMS pulse by providing a trigger signal, and data was collected at 5 µs intervals by adjusting the time delay of this pulse. The time-of-flights of $Ar^+$, $N^+$, $Al^+$ , $Cu^+$ and $W^+$ ions measured in this way are compared to those calculated using mass spectrometry flight tube equations.

**PP-ThP-5 Focused Magnetron Sputtering: A Comprehensive Study of Magnetron Power Effects on AlCrN Coatings Under Industrial Conditions**, ***Martin Ucik [m.ucik@platit.com]***, Masaryk University, Czechia

**Introduction**

Traditional coating methods, such as Cathodic Arc Evaporation (CAE), face challenges due to microscopic defects and other limitations. Focused Magnetic Field Magnetron Sputtering (F-MS) has emerged as a transformative solution, achieving a high ionized metal flux fraction even for large-scale targets [1]. Compared to conventional magnetron sputtering (DCMS), F-MS demonstrates a six-fold increase in power density [2]. This advantage, combined with effective cooling and prolonged duty cycles, establishes F-MS as a groundbreaking technology. Its integration into PLATIT's PVD coating unit, Pi411, represents a significant advancement in hard protective coatings for industrial applications.

**Methods**

F-MS operates by moving a reduced-size magnetron longitudinally inside a tubular target (Ø110 × 510 mm). This design enables high-power sputtering of up to 30 kW and allows the deposition of dense coatings at a growth rate of 2 µm/h using a 3-fold carousel rotation system.

**Results**

Coatings of (Al,Cr)N deposited via F-MS exhibited stoichiometric composition, smooth surfaces, and controlled defect levels. Mechanical property tests, plasma diagnostics, and cutting tests demonstrated strong interrelationships and benefits associated with higher power levels. Notably, cutting tests confirmed the superior performance of (Al,Cr)N coatings compared to state-of-the-art CAE coatings.

**Conclusion**

F-MS technology represents a significant breakthrough in the coating industry, addressing the limitations of traditional methods. Its ability to achieve high plasma power densities and a high degree of ionization for large-scale targets holds immense potential to advance industrial coating practices by enhancing efficiency and enabling new applications.

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[2] Klimashin, F. (2023). High-power-density sputtering of industrial-scale targets: Case study of (Al,Cr)N. Mat. & Des., 237, 112553. https://doi.org/10.1016/j.matdes.2023.112553

**PP-ThP-7 Energy Flux Diagnostics in High Power Impulse Magnetron Sputtering**, ***Caroline Adam [c.adam@physik.uni-kiel.de]***, Kiel University, Germany*; Holger Kersten,* Kiel University, Kiel Nano, Germany

High power impulse magnetron sputtering (HiPIMS) has shown significant potential for thin film deposition. This potential is evident through the enhancement of film quality, specifically in terms of increased density [1] and adhesion [2] along with the diminished requirement for high substrate

temperatures [3]. To achieve the optimal deposition process, it is crucial to develop a comprehensive understanding of the plasma-surface interaction at the substrate. This includes, in particular, analyzing the energy flux (transferred power from the plasma to the surface) and its composition.

The energy flux is investigated by using a passive thermal probe (PTP) [4], a so-called non-conventional diagnostic, measuring the integral energy flux to the substrate. Insights into the composition of the energy flux are gained by applying a bias voltage to the thermal probe [4] and using a novel combination of PTP with a retarding field analyzer (RFA) [5]. This allows to measure simultaneously the ion energy distribution (IED) and to perform energy-resolved energy flux measurements. In addition, the neutral energy flux component can be quantified by repelling all charge carriers by the grid potentials. Since the energy resolution and sensitivity of the RFA is limited, the measurements of the IED are completed by energy-resolved mass spectrometry, both time-averaged and time-resolved [6].

These diagnostics have been applied to compare HiPIMS and DC magnetron sputtering processes with same gas pressure and average power sputtering a planar copper target in argon atmosphere. In total, the mean energy flux to the substrate is lower in HIPIMS operation. Hence, temperature sensitive substrates are better protected. Normalizing the energy flux to the deposition rate, which is lower in HiPIMS as well, gives a higher value for the energy flux per adatom in HiPIMS, which can be attributed to the higher kinetic energy of sputtered particles. The dependence of the energy flux on the excitation mode (DC, HiPIMS), the HiPIMS pulse parameters, as well as on power and pressure is investigated. The advantages and limitations of the diagnostics used are discussed.

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[2] R. Bandorf et al., Surf. Coat. Technol. 290 (2016) 77-81.

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[6] J. Benedikt et al., J. Phys. D: Appl. Phys. 45 (2012) 403001.

**PP-ThP-8 Enhancement of Barrier Properties of Aluminum Oxide Layer by Optimization of Plasma-Enhanced Atomic Layer Deposition Process**, ***Hyun Gi Kim [opti_people@khu.ac.kr]***, KyungHee University, Republic of Korea

Aluminum oxide (AlxOy) layers were deposited on polyethylene naphthalate substrates by low frequency plasma-enhanced atomic layer deposition process for barrier property enhancement. Trimethylaluminum and oxygen plasmawere used as precursor and reactantmaterials, respectively. In order to enhance the barrier properties several process parameters were examined such as plasma power, working pressure and electrode–substrate distance. Increase of plasma power enhanced the reactivity of activated atomic and molecular oxygen to reduce the carbon contents in $Al_xO_y$ layer, which appeared to enhance the barrier properties. But too high power caused generation of byproducts which were reincorporated in AlxOy layer to reduce the barrier properties. Plasma generated at lowerworking pressure was providedwith an additional energy for reactions and hadmore diffusion of the plasma. The O/Al ratio of the layer approached the stoichiometric value by increasing the electrode–substrate distance. At the following conditions: 300 W of plasma power, 26.7 Pa of working pressure and 50 mm of electrode–substrate distance, water vapor transmission rates of the AlxOy layer reached $8.85 \times 10^{-4}$ g/m$^2$ day.

**PP-ThP-9 Development of DC Magnetron Sputtered Ni-Fe Bimetallic Thin Film Anodes for Oer in Water Electrolysis for Hydrogen Production and Optimization of Composition for Ni-Fe Bimetallic System**, *Daniyal Hasan,* ***Sandra Carvalho [sandra.carvalho@dem.uc.pt]***, *Albano Cavaleiro, Diogo Cavaleiro,* University of Coimbra, Portugal

Hydrogen production through water electrolysis provides clean source of energy and the process has a zero-carbon emission cycle. Different performance parameters have been focused to improve the hydrogen production but equally important is the oxygen evolution reaction at the anode side which is the other half cell reaction in the process. Half-cell reaction at anode side has the problems of slower reaction kinetics, complex chlorine oxidation, cost and the degradation of electrode materials like $RuO_2$/$IrO_2$, and low current densities which do not meet industrial requirements.

We have used DC magnetron sputtering to deposit Ni-Fe bimetallic thin films over SS-316 substrate as efficient and cost-effective anode material. Ni-Fe bimetallic compounds have been reported to be used in this type of process but unlike the traditionally reported wet chemical synthesis techniques, we have used PVD technique to have superior control over micro structure for better performance and to avoid chemical waste.

In a novel effort we have deposited thin films with 200 nm thickness through DC magnetron sputtering. Thin targets of Invar and Ni with 1 mm thickness were sputtered under 10 bar Argon pressure to minimize the paramagnetic behavior of Ni and Fe. Different compositions were achieved by varying the sputtering power on Ni target from 500-1200 W while Invar was sputtered at fixed power of 1000 W. Compositions were achieved in the range of 20-40 atomic % Fe in Ni which were analyzed through Energy Dispersive Spectroscopy (EDS). Scanning Electron Microscopy (SEM) analysis showed that films had columnar structure which increases the available surface area. Atomic force microscopy (AFM) analysis showed very low roughness of the films in the range of 1-2 nm. X-Ray diffraction (XRD) analysis showed the presence of $FeNi_3$ phase which has an efficient performance in the reversible oxidation-reduction reactions at anode.

Electrochemical testing techniques including linear sweep voltammetry (LSV), cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) were performed in a three-electrode cell with Ag/AgCl saturated in KCl reference electrode, graphite counter electrode and 1 M KOH electrolyte. Composition of $Fe_{24}Ni_{76}$ (24 atomic % Fe in Ni) was found to be the optimum composition as it achieved remarkable current density of 335 mAcm$^{-2}$ while operating at overpotential of 1.7 mV vs RHE which is the benchmark overpotential beyond which Chloride oxidation becomes thermodynamically possible. EIS analysis showed that $Fe_{24}Ni_{76}$ composition has the least charge transfer resistance ($R_{CT}$) with uniformity and behavior close to an ideal capacitor.

**PP-ThP-10 Exploring Green Alternatives for Plasma Etching of Silicon Carbide**, ***Chang-Koo Kim [changkoo@ajou.ac.kr]***, *Sanghyun You,* Ajou University, Republic of Korea

Silicon carbide (SiC) has a wider band gap enrgy than silicon (Si), enabling efficient operation in high-temperature environments. Its strong Si-C bonds also provide exceptional physical and chemical stability. These properties make SiC an ideal material for power semiconductor devices, including insulated gate bipolar transistors (IGBTs), Schottky barrier diodes, and metal-oxide-semiconductor field-effect transistors (MOSFETs).

SiC etching is traditionally performed using $SF_6$, $CF_4$, and $CHF_3$ plasmas. However, these gases have stable molecular structures, leading to high global warming potentials (GWPs) and long atmospheric lifetimes. Recognized as greenhouse gases under the Kyoto Protocol, their emissions are subject to reduction targets outlined in the Paris Agreement. To minimize environmental impact, there is a growing need for low-GWP alternatives in SiC etching.

This study explores the use of heptafluoroisopropyl methyl ether (HFE-347mmy) as a plasma etchant for SiC. With a GWP of approximately 530, HFE-347mmy significantly reduces environmental impact compared to $SF_6$, $CF_4$, and $CHF_3$. The etching characteristics were investigated by varying the HFE-347mmy/$O_2$ flow-rate ratio and adjusting the bias voltage. Additionally, radical concentrations, surface composition, and substrate roughness were analyzed to elucidate the etching mechanism.

**PP-ThP-11 Optical Emission Spectroscopy Signal Analysis for Predicting Deposition Characteristics of Silicon Nitride in Plasma Enhanced Chemical Vapor Deposition**, ***Youngju Ko [kyj900903@naver.com]***, *Hyeonjin Choi, Jinmyeong Kim, Namgun Kim, Heeyeop Chae,* Sungkyunkwan University (SKKU), Republic of Korea

Optical emission spectroscopy (OES) is a non-invasive tool that enables plasma monitoring without affecting the plasma state. It operates by analyzing light emitted from excited atoms or ions within the plasma. This analysis provides information about the chemical composition and energy state of the plasma thus allowing for the prediction of process results. Researchers have conducted studies analyzing OES signals during deposition process to predict deposition characteristics such as growth rate, film thickness, uniformity. However, research on predicting the refractive index, which is one of the important deposition characteristics, has been limited. In this study, the deposition rate and refractive index of silicon nitride ($SiN_x$) were predicted using OES signal analysis in plasma enhanced chemical vapor deposition (PECVD) with trisilylamine (TSA), $NH_3$, and $N_2$ gases. The deposition rate was correlated with the $I_{N2+}/I_{N2}$ and $I_{Ha}/I_{Hb}$ line ratio, which can estimate the electron temperature, as the deposition rate is influenced by the electron temperature that activates dissociation and ionization. The refractive index determined by the N/Si ratio in $SiN_x$ film was correlated with the ratio of SiH and NH radicals in the plasma transferred to the film. The relative radical densities were investigated using

the $I_{SiH}/I_{N2}$ and $I_{NH}/I_{N2}$ line ratios, known as actinometry, after demonstrating the overlap of electron energy distribution function (EEDF) and excitation cross sections. The deposition rate ($R^2$ = 0.85, MAPE = 3.66%) and refractive index ($R^2$ = 0.95, MAPE = 0.27%) investigated in linear regression analysis showed very high prediction accuracy.

**PP-ThP-12 Surface Engineering for the Interface between p-Type Germanium and Alloy-Like Hafnium Nitride Buffer Layer with Pre-Hydrogen Plasma Trimming**, ***Bo-Syun Syu [brian20000713@gmail.com]***, National Tsing Hua University, Taiwan*; Dun-Bao Ruan,* Fuzhou University, China*; Kuei-Shu Chang-Liao, Hsin-I Yeh,* National Tsing Hua University, Taiwan

A pre-hydrogen plasma trimming treatment was applied on p-type germanium (Ge) metal oxide semiconductor device as a surface engineering process, which significantly improve the interface quality between p-type Ge and alloy-like hafnium nitride buffer layer. Generally, a post interfacial layer treatment is difficult to affect the defect distribution located at the bottom interface. Therefore, a pre-plasma treatment is well discussed in this work. After detailed analyzing material and electrical characteristics, the sample after the pre-hydrogen plasma trimming treatment exhibits lower equivalent oxide thickness, lower interface trap density, narrower frequency dispersion, better uniformity and reliability. This improvement can be attributed to the reduction of both border traps and surface roughness. It is believed that this research will provide an important reference for the high mobility Ge based device fabrication.

**PP-ThP-13 Chamber Design and Capabilities for Nanocalorimetry-Based Plasma Diagnostics**, ***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

There is an urgent need to expand standard plasma diagnostics methods for a faster and comprehensive description of plasma-surface processes associated to nanofabrication routines, such as film deposition and surface etching or cleaning. In a recent study [Diulus et al, J. Vac. Sci. Technol. B 43, 020601 (2025)], the flux of atomic radicals from a hydrogen discharge has been quantified using differential nanocalorimetry in cold plasma environment. This approach is novel and motivates the development of a new plasma probe, based on nanocalorimetry principle, which should go beyond plasma thermometry by effectively interrogating plasma parameters as well as basic plasma-surface interaction processes. Here, we report on the design and capabilities of a research plasma reactor equipped with state-of-the-art plasma diagnostics devices aimed to benchmark the thermal data provided by the nanocalorimeter. The high-vacuum chamber consists of a six-ports cross fed by a remote inductively coupled plasma (ICP) source with adjustable plasma plume position. The nanocalorimeter sensor is installed along with the following instruments: (1) Langmuir probe to provide plasma parameters and electron energy probability function (EEPF); (2) sensor combining a retarding field energy analyzer (RFEA) and a built-in quartz microbalance to evaluate ion energy distributions and ion-to-neutral flux ratios, and (3) optical emission spectroscopy (OES) together with (4) quadrupole mass spectrometer to survey the chemical composition of the analyzed plasma. Sensors (1), (3), and (4) provide information from the plasma bulk, while probe (2) collects fluxes of plasma species at the sheath level in contact with substrate/electrode. A few plasma nanocalorimetry platform parameters, such as sensitivity, response time, and stability, will be reported. Once optimized, the nanocalorimetry platform will be expected to monitor surface modification processes by reactive plasmas with unparalleled response times and sensitivities within an extended range of particle fluxes and densities.

**PP-ThP-15 Magnetron Sputter Deposition of Corrosion Resistant Thin Films on Al Surfaces**, ***Tomas Kubart [tomas.kubart@angstrom.uu.se]***, *Yao Yao,* Uppsala University, Solid State Electronics, Sweden*; Karin Törne, Smita Rao, Hannes Nedersted, Live Mölmen, Anders Lundblad,* RISE Research Institutes of Sweden

Aluminium is an attractive material for bipolar plates for polymer electrolyte membrane fuel cells due to its low weight, excellent thermal and electrical conductivity, as well as good recyclability. However, to achieve the necessary low contact resistance and corrosion stability, a suitable surface coating is needed. We could previously show that the corrosion current can be significantly reduced from 20 to 0.2 $\mu A \cdot cm^{-2}$ by coating the Al surface with a thin film of Ti. The coated surfaces, however, exhibited limited durability during potentiostatic testing because of a large number of defects in the Ti layer.

This contribution analyses formation of defects in Ti thin films deposited by magnetron sputtering. Cross-sectional analysis revealed that these defects are initiated already at the Al/Ti interface and enhanced during the deposition. Ti growth by High Power Impulse Magnetron Sputtering and by direct current Magnetron Sputtering on different Al surfaces and on Si is investigated to identify the effect of the surface and growth conditions on the film growth.

It is shown that segregated secondary phases in the Al substrate as well as surface imperfections may act as initialization points for the defect formation. Further, mitigation strategies to improve the film quality are discussed.

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