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

PP-ThP-1 Optimizing Thin Film Deposition with Ion Energy and Flux Measurements in Pulsed Plasmas with Plasma Diagnostics*, **Angus McCarter [angus.mccarter@impedans.com]**, Thomas Gilmore, Anshu Verma,* Chase House, City Junction Business Park, Ireland

In the field of metallic coatings and thin film deposition, precise control of deposition processes has become crucial. These processes are heavily dependent on the chemical/physical processes occurring on the substrate surface. Like any other surface in contact with plasma, a sheath usually develops on the substrate which pulls down the ions out of bulk plasma necessary to complete the process on the substrate. Furthermore, external RF/DC/tailored waveform biases are applied to the substrate to modify the ion behaviors. As it can affect the chemical composition, microstructure and the associated electrical properties of the thin films during plasma assisted deposition processes. Therefore, the characterization of only bulk plasma is not sufficient in providing insights necessary to understand the plasma surface interactions. A high-speed monitoring of the ion energy distribution function and ion flux can lead to enhanced understanding of the plasma surface interactions and improved process performance.

We will highlight the successful measurements done by the Semion RFEA diagnostic under different chamber and bias conditions. Such applications enabling accurate and precise control of deposition process on different materials and various plasma chemistries. It measures the ion energies hitting a surface, the ion flux, negative ions and bias voltage at any position inside a plasma chamber using an array of integrated sensors. On the other hand, the Semion pDC system measures these parameters in real time over an energy range up to 2000 eV (process dependent). It can do sub-microsecond time resolved measurements, for studying pulsed ICPs, or pulsed DC biases, as well as floating and grounded substrate conditions. The Semion Pulsed DC system is the key instrument used to measure the temporal evolution of the ion energy and flux at different times through the pulse period of a pulsed DC plasma process.These measurements are essential for establishing the correlation between the plasma inputs and the ion energy/flux which, in-turn, determines the effectiveness of the surface treatment.

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-6 Design and Evaluation of a Laboratory-Scale Thermal ALD Reactor: Case Study with Aluminum Oxide and Zinc Oxide.*, **Jackeline Navarro-Rodriguez [jackeline.navarro@uabc.edu.mx]**, David Mateos-Anzaldo, Jesus Martinez-Castelo, Rogelio Ramos-Irigoyen, Oscar Perez-Landeros, Mario Curiel-Alvarez, Benjamin Valdez-Salas,* UNIVERSIDAD AUTONOMA DE BAJA CALIFORNIA, Mexico*; Eduardo Martinez-Guerra,* CIMAV-Monterrey, Mexico*; Hugo Tiznado-Vázquez,* UNAM, Mexico*; Nicola Nedev,* UNIVERSIDAD AUTONOMA DE BAJA CALIFORNIA, Mexico

Atomic layer deposition (ALD) is a crucial technique in microelectronics for thin film deposition due to its precise thickness control at the atomic scale, although its sustainability remains a challenge due to its high cost. This work presents the results of a customized, cost-effective and efficient thermal ALD system designed to deposit semiconducting and insulating thin films such as aluminum oxide (Al_2O_3) and zinc oxide (ZnO) films, using trimethylaluminum (TMA) and diethylzinc (DEZ) as precursors, respectively. The oxidizing agent used was hydrogen peroxide (H_2O_2).

Thin films of Al_2O_3 and ZnO were deposited on silicon and corning glass substrates at a temperature of 200°C. Ellipsometry measurements were carried out to determine the thickness, optical constants, band gap and transparency.

The results obtained indicate the presence of a thin film deposited on silicon with 1160 cycles, with a growth rate of 1 Å/cycle which is equivalent to a thickness of ~116.4 nm, as well as a refractive index of 1.76, an extinction coefficient of 0. In addition, the zinc oxide film deposited on corning glass present approximately 80% of transparency in visible region.

The system stands out for its optimized design, easy handling and low cost, which makes it a viable option for academic research and applications in electronics and nanotechnology.

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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