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

PVD Coating Technologies I

Moderators: Christian Kalscheuer, RWTH Aachen University, Germany, **Vladimir Pankov**, National Research Council of Canada

8:00am **PP1-1-TuM-1 Discharges Modes Relevant to Plasma-Based Coatings: an Analysis of Their Physics and Economics***, Andre Anders* ***(andre.anders@iom-leipzig.de)***, Leibniz Inst. of Surface Eng. (IOM), Germany **INVITED**

Physical Vapor deposition (PVD) has matured over the last decades, where plasma processes have been added to control the microstructure of thin films and coatings. The different processes make use of different discharge modes, such as magnetically enhanced glow (magnetron discharge) and arc discharges, where the latter has the most prominent and very different sub-modes of thermionic and cathodic arcs.However, these are not the only ones.The overview connects the discharge physics of the different modes – resulting in plasmas of quite different properties – to the film microstructure, control options, but also considers the economics (especially energy considerations) associated with the different approaches.

8:40am **PP1-1-TuM-3 Design of an Innovative Cathodic Arc Source with High Deposition Rate and Low Macroparticles Generation***, Raül Bonet* ***(raul.bonet@eurecat.org)***, Eurecat Technological Center of Catalonia, Spain*; L. Carreras,* Tratamientos Térmicos Carreras S.A, Spain*; J. Orrit-Prat, J. Caro,* Eurecat Technological Center of Catalonia, Spain

Within the group of PVD techniques, the cathodic arc evaporation technique stands out for its performance, which is characterized by a nearly 100% ionized, high energy plasma. Its versatility, lower economic cost compared to other deposition techniques and easy industrial scaling has led to a massive industrial implementation for the preparation of coatings and thin layers of different nature. One of the disadvantages of the cathodic arc technique is the generation of macroparticles because of the electric arc discharge on the cathode surface. This fact can be an important technological limitation for those cases where a good surface finish, low friction coefficient or good wear and corrosion resistance is required. Traditionally, this problem has been solved by applying magnetic filters that separate the macroparticles from the plasma ions. However, this solution involves a considerable reduction in the deposition rate, which makes its industrial implementation difficult. On the other hand, in order to reduce the coating process costs, cathodic arc sources that allow a high deposition rate are required.

In this work, an innovative cathodic arc source has been designed to achieve a deposition rate of up to 20 microns/hour, with a reduction of 80 % of macroparticles. In order to increase the deposition rate, a high-current pulsed source (100-500 A, 10-20000 Hz) has been used to generate the arc discharge between the anode and cathode. On the other hand, to reduce the generation of macroparticles, an optimized magnetic field configuration around the cathode has been obtained by means of Finite Elements Method (FEM) simulation, which allows to induce a fast and homogeneous movement of the electric arc on the cathode surface. Standard transition metal nitride (CrN, AlTiN) hard coatings obtained using this source exhibit excellent surface finish and improved mechanical properties in terms of adhesion and hardness.

9:00am **PP1-1-TuM-4 TaB_X Thin Film Synthesis from an Industrial-Sized DC Vacuum Arc Source***, Igor Zhirkov* ***(igor.zhirkov@liu.se)****, A. Petruhins, A. Shamshirgar,* Materials Design Division, IFM, Linköping University, Sweden*; S. Kolozsvári, P. Polcik,* PLANSEE Composite Materials GmbH, Germany*; J. Rosen,* Materials Design Division, IFM, Linköping University, Sweden

Thin films of transition metal borides are gaining increasing attention due to their physical and chemical characteristics. Most publications in this area focus on TiB_2, synthesized through various sputtering techniques, targeting applications as protective hard layers. Tantalum diboride, TaB_2, is another system with interesting properties, especially for high temperatures, but is much less explored. The elastic modulus of TaB_2 is ~ 2 times lower than that of TiB_2, but displays similar high hardness, combining high strength with high resistance to elastic and plastic deformation. Deposition of TaB_2 coatings with the industrially relevant physical vapor deposition (PVD) process DC vacuum arc is virtually absent in the literature. Still, DC arc deposition allows synthesis of coatings with a deposition rate unreachable for any other PVD technique. This motivates development and investigation of arc processes for TaB_2 synthesis. In the present work, we investigate DC arc plasma generation and deposition of TaB_x, and compare to previously investigated TiB_x. We use an industrial scale arc plasma source, Hauzer CARC+, which utilizes plane cathodes of 100 mm in diameter. Process stability and cathode dependent features of arcing is evaluated, and plasma analysis with respect to charge-state resolved ion energy is performed, showing a high ionization degree, and ion energies extending well above 100 eV. It is well known that plasma generation from compound cathodes gives a mass-dependent angular distribution for the elements of the compound, which is confirmed for the here investigated borides. This, in turn, is one of the factors contributing to a resulting film composition diverging from the cathode composition. The plasma characterization and macroparticle generation is correlated to deposited thin films; their composition, structure and properties. Altogether, the results show that DC vacuum arc is an industrially relevant technique for deposition of metal diborides.

9:20am **PP1-1-TuM-5 Plasma Enhanced Magnetron Sputtering and Its Applications in Industry***, Jianliang Lin (jlin@swri.org)***, Southwest Research Institute, USA **INVITED**

Plasma enhanced magnetron sputtering (PEMS) technology is an advanced version of the conventional magnetron sputtering technique. The PEMS technique draws electrons off of hot filaments installed in a sputtering system when electrons have gained enough energy to exceed the work function of the filaments. The electrons collide with neutral atoms and generate a large number of ions through impact ionization. As a result, a global hot filament assisted plasma is formed in the entire chamber which is independent of the magnetron discharge plasma.The hot filaments also provide additional thermal energy without using external heating elements. Plasma diagnostics showed that the majority of the ions in the PEMS plasma exhibited low energies of less than 5 eV. However, a significant increase in the ion flux can be achieved by increasing the hot filament discharge current. The extra ion fluxes provide enhanced ion bombardment on the substrates, which is beneficial for improving the structure and properties of coatings. The PEMS plasma can be utilized to perform different surface engineering tasks, e.g. plasma cleaning/etching, plasma nitriding, and coating depositions. It can be easily combined with other magnetron sputtering techniques, e.g. DC, RF, pulsed DC, and high power impulse magnetron sputtering (HiPIMS) to enhance ion fluxes and thermal energies. In this presentation, the principle and characteristics of the PEMS technology will be introduced. Technical examples of PEMS coatings for different industry applications will be reviewed, for example, solid particle erosion resistant coatings for aerospace and Oil&Gas, duplex coatings for die casting dies, low friction nanocomposite coatings for combustion engine piston rings, protective coatings for high temperature sCO_2 environment, etc.

10:00am **PP1-1-TuM-7 Sustainable and Economical Production of High-Quality HIPIMS Coatings***, Stephan Bolz (stephan.bolz@cemecon.de)****, B. Mesic, O. Lemmer, C. Schiffers,* CemeCon AG, Germany

Constant improvement of ceramic coatings for cutting tools aiming at best wear resistance under conventional and extreme conditions is driven by the development of new workpiece materials with improved properties. Economical machining of such materials requires ever denser and harder coatings with better adhesion to the tool substrate. In addition to the required coating properties, however, the economical production of these coatings plays a more important role since some time. Shorter coating processes, reduced handling and lower energy consumption are the right keywords to well describe the current situation.

Considering these aspects, high-performance coating technologies, such as HiPIMS, are becoming more and more interesting for the market. Thanks to HiPIMS dense, hard, adhesive, and droplet-free layers can be deposited in highest quality with high energy efficiency at high deposition rates. Furthermore, well-chosen HiPIMS pulse parameters combined with an appropriate bias synchronization can avoid high residual stress of coatings for sharp edged cutting tools.

In our presentation we show that optimization of HiPIMS pulse parameters leads to a significant increase in metal ionization, accompanied by improved coating properties of an (Al,Ti,Si)N layer. The improved coating properties include above all a denser microstructure and a smoother surface, which allows to skip time consuming and energy-intensive post-treatment steps. Brilliant shine and best optical appearance are related with low friction and perfect chip removal during use. This combination of layer properties is a guarantee for a perfect surface finish of the workpiece.

10:20am **PP1-1-TuM-8 Increasing the Metal Ion Flux Fraction in Industrial Conditions**,
Peter Klein (pklein@mail.muni.cz), *J. Hnilica,* Masaryk University, Czechia*; V. Sochora,* SHM s.r.o., Czechia*; P. Vašina,* Masaryk University, Czechia

The field of coating technologies progressively changes. Before, PVD technologies were utilized to produce a coating with a single superior quality such as very high hardness, high ductility, or very low roughness. Nowadays, due to increasingly sophisticated applications, a combination of superior properties is required. Generally, high-quality coatings require high ion flux to be formed, which is easily achievable by arc–based techniques, but those are prone to have high roughness of produced films. This work reports on high ionized metal flux fraction (IMFF) in industrial DC magnetron conditions.

A biased QCM was employed in the industrial magnetron deposition system to quantify the metal ion contribution to the forming film. The used deposition system can utilize up to four cylindrical rotating electrodes, each measuring 50 cm in length and 10 cm in diameter. For the titanium magnetron cathode, up to 40 kW DC power can be supplied over the 90 cm^2 racetrack, providing a current density of around 0.8 $A.cm^{-2}$. Such a high current density is rather similar to pulsed power techniques and unsurprisingly, up to 30% of IMFF can be obtained purely in the DC operation. A striking difference from laboratory DC magnetron discharges, where the literature states the IMFF can reach only up to 2%.

The IMFF can be further enhanced in the described system through the utilization of a lateral glow discharge (LGD). Lateral glow discharge uses one of the arc electrodes as an electron source. Produced electrons from the arc cathode are drawn to the opposite arc electrode, forming an area over the magnetron cathode where the collision with the sputtered metal occurs. This increases IMFF by a further 10%, which makes this a comparable system to HiPIMS. Unlike HiPIMS, the introduction of LGD into the DC magnetron sputtering process does not affect the deposition rate. Despite LGD being arc-based technology, it does not create macroparticles, but It has to be noted that the LGD requires multi-electrode configuration to run.

10:40am **PP1-1-TuM-9 Unraveling the Dynamics of Reactive Magnetron Sputtering: Insights into Feedback Control, Metastable Conditions, and Long-term Stability**,
Josja Van Bever (Josja.VanBever@UGent.be), *K. Strijckmans, D. Depla,* Ghent University, Belgium

High-quality coatings with optimized stoichiometry in reactive magnetron sputtering are essential for numerous industrial applications. This demands meticulous control of process conditions within the transition region between metallic and poisoned modes, achieved through feedback control or high pumping speeds [1]. Despite advancements, a comprehensive understanding of the convergence and steady-state conditions in feedback control for this context is still elusive.

In this study, we present *precise feedback* measurements, focusing on the intricate dynamics within the aluminum-oxygen system. Our investigations unveil two distinct metastable states dependent on the system's history. We establish a clear connection between these states and the *double hysteresis* phenomenon, as predicted by the Reactive Sputtering Deposition (RSD) model [2-4] and substantiated by prior research on IV-characteristic data analysis [5]. This linkage is achieved through careful manipulation of discharge current density and process parameters associated with target poisoning mechanisms.

Delving into the *long-term stability* of the double hysteresis in feedback control, we compare it with stable transition conditions achieved through high-pumping speeds. Our discussion encompasses various factors influencing *long-term stability, including target erosion effects [6], chamber wall gettering, anode effects [7]*, and fluctuations induced by the chosen process control. *A new type of feedback input signal is introduced* to compensate for plasma and chamber wall effects.

Furthermore, we explore diverse feedback convergence strategies, shedding light on the path toward an optimal approach that considers the stability of each set of transition conditions. This understanding provides a valuable guideline for industry professionals seeking to employ feedback control reproducibly and efficiently.

Our research significantly contributes to the body of knowledge in reactive magnetron sputtering, providing insights into the intricate interplay between feedback control, metastable conditions, and long-term stability. These findings promise to elevate the precision and reliability of thin film deposition processes, with implications for a wide range of technological applications.

References are found in the supplementary material.

Author Index

Bold page numbers indicate presenter