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

PVD Coatings and Technologies I

Moderators: Dr. Qi Yang, National Research Council of Canada, **Dr. Christian Kalscheuer**, IOT, RWTH Aachen, Germany

10:00am **PP1-1-MoM-1 Complementary Cutting-Edge Plasma Monitoring Techniques for Process Development, Production Control and Machine Learning (ML)**, ***Thomas Schütte [schuette@plasus.de]***, *Jan-Peter Urbach, Peter Neiß, Marius Radloff, Hokuto Kikuchi,* PLASUS GmbH, Germany **INVITED**

As specifications in the thin film industry become more and more demanding, high production yields and cost effective production becomes a major factor in this competitive market. Increasing demands for better specifications and lower scrap rates drive the demand for efficient process control systems.

In addition, data analysis using artificial intelligence (AI) and machine learning (ML) technologies has made tremendous progress in recent years, sparking interest in using these methods for the diagnostics and control of plasma applications. To utilize this capability, a large number of data sets from complementary process diagnostics methods are required.

This presentation will highlight the opportunities and advantages of utilizing the latest developments in real-time in-situ data acquisition of different diagnostic techniques in a single system: Spectroscopic plasma process monitoring acquires data from the actual process plasma whereas in-situ broadband photometric measurements gather properties of the growing coating such as film thickness or color values. In addition, time-resolved electrical measurements of generator power, voltage and current provide valuable electrical process information especially in pulsed plasma applications.

Selected plasma applications are used to illustrate how process variations influence the results of the different measurement techniques. Consequently, by combining different methods and analyzing the complementary data in real-time, interdependencies between process and product properties become visible and can be used to achieve more accurate and reliable process control. At the same time the collected data can be fed into the analysis using AI and ML techniques to improve product quality and long-term production stability.

Real-time process control examples combining different diagnostic methods will be presented and first approaches to the application of ML methods will be illustrated using various coating applications from industry and R&D, such as metallic and reactive sputtering, HIPIMS and PECVD processes for tribological, optical and glass coating processes.

10:40am **PP1-1-MoM-3 Plasma Diagnostics and Thin Film Synthesis Using an Industrial-Sized DC Vacuum Arc Source with Magnetic Steering and a TaB_2 Cathode**, ***Igor Zhirkov [igor.zhirkov@liu.se]***, *Andrejs Petruhins, Ali Saffar Shamshirgar,* Materials Design Division, Linköping University, Sweden*; Philipp Immich,* IHI Hauzer Techno Coating B.V., Netherlands*; Szilard Kolozsvári, Peter Polcik,* PLANSEE Composite Materials GmbH., Germany*; Johanna Rosen,* Materials Design Division, Linköping University, Sweden

Due to physical and chemical characteristics of transition metal borides, thin films thereof are gaining increasing attention as protective hard coatings. Most publications in this area focus on TiB_2, synthesized through various physical vapor deposition (PVD) techniques. Tantalum diboride, TaB_2, is characterized by a hardness comparable to TiB_2, but with an elastic modulus ~ 2 times lower. It results in a combination of high strength and high resistance to elastic and plastic deformation, for potential use in protective coating industry. However, there are no reports on deposition of TaB_2 coatings with DC vacuum arc, a process commonly used in industry, in particular with magnetic steering. In this work, we present analysis of the process (cathode weight loss, film deposition rate), plasma composition, and the (micro-) structure and composition of the cathode as well as of films, using a Hidden EQP mass-energy analyzer, SEM, XRD, and XPS. The study is performed using an industrial scale arc plasma source, Hauzer CARC+, which utilizes plane cathodes of 100 mm in diameter. The TaB_2 cathodes were provided by PLANSEE Composite Materials GmbH. The magnetic arc steering system, based on variation of magnitude of electrical current flowing through the solenoid placed behind the cathode, allows tuning of the strength of the magnetic field at the center point of the cathode surface in the range ± 8 mT (different polarity). The steering system is found to improve the stability of the arcing process and result in more smooth erosion of the cathode surface. Plasma analysis performed at base pressure (10^{-5} Torr) shows peak ion energies consistent with the velocity rule, around 140 and 10 eV for Ta and B, respectively. The ion energies, the ion charge states, and the plasma ion compositions were found to be strongly affected by the operating pressure, with a plasma ion composition showing a lower B content (~ 60 % Ta and ~ 40 % B at base pressure) compared to the cathode stoichiometry. Even lower B ion signals were recorded at higher pressures. The plasma properties were correlated to the deposited thin films, their composition and structure. The lack of B within the deposited films was found to be less pronounced than for the plasma ions. Altogether, the results show that DC vacuum arc can be used for TaB_2 depositions with stability provided by the magnetic steering.

11:00am **PP1-1-MoM-4 Novel Approach in Cathodic Arc Evaporation Enabling Precise Control Over Energy of Deposited Ions in Industrial Conditions**, ***Martin Ucik [m.ucik@platit.com]***, Masaryk University, Czechia

Introduction

Commonly, in cathodic arc evaporation (CAE), where multiple elements are contained in a coating, materials of several targets are simultaneously evaporated. This evaporated material, being near-complete ionization due to the exceedingly high power densities in cathode spots with significant number or even predominance of multiply charged ions (2+, 3+, etc.), is condensed onto the substrate under applied bias which is constant. Therefore, often the currents and the substrate bias need to be optimized in order to avoid undesirable effects such as delivering excessive energy to the growing coating.

With a new approach of pulsed arc and synchronized bias relative to the arc pulses, we are now able to select which evaporated material is accelerated towards the substrate at what applied voltage, therefore controlling the delivered energy or, in other words, having a mechanism to regulate the energy of impacting ions.

Methods

Here, we choose one of the most widely studied coatings – AlCrN – to investigate the effect of synchronized bias. On two single element targets (Al, Cr), both arcs are simultaneously periodically pulsed and a substrate bias (from 30V up to 240V) is either constant or also pulsed in respect to the arc pulses (e.g. by Al pulse, we mean that arc current of Al cathode is at its maximum level). This offers us three basic modes we have focused on: (i) with a constant substrate bias, (ii) with bias applied during the Al pulse and leaving the substrate at a floating potential during Cr pulse and (iii) vice-versa applying the bias only during Cr pulse.

Results

By simply choosing the mode other than (i), we can change the coating microstructure, thus the properties – e.g., grain size, lattice orientation, residual stress, etc. We also investigated the effect of synchronized bias in a cutting test, where we observed an influence of a mode choice on cutting edge wear.

Conclusion

In summary, this study shows new possibilities of cathodic arc evaporation enabling to fine-tune coating microstructure to obtain desired properties and further more cutting performance.

to be submitted to Technical Symposium PP Plasma and Vapor Deposition Processes, and section PP1 PVD Coatings and Technologies (oral contribution)

11:20am **PP1-1-MoM-5 Industrial-Scale PVD Deposition of Aluminium Oxide**, ***Ivan Kolev [ikolev@hauzer.nl]***, IHI Hauzer Techno Coating B.V., Netherlands*; Philipp Immich, Daniel Barnholt, Julia Janowitz, Louis Tegelaers,* IHI Hauzer Techno Coating B.V.,, Netherlands*; Rolf Schäfer,* Robeko GmbH & Co. KG, Germany*; Tobias Radny,* Robeko GmbH & Co., KG, Germany

Aluminium oxide (Al_2O_3) is a well-known material with versatile properties, such as high hardness, electrical insulation, chemical inertness, and thermal stability. These properties make alumina also a desired thin film in various industrial applications. Its hardness and thermal stability are very beneficial for coatings on inserts. Other emerging application field is the sensor coatings, where the need ofhighly insulating films makes aluminium oxide a primary choice. Its optical transparency in combination with high hardness and chemical resistance find application in protective coatings.

Despite these multiple applications, efficient industrial-scale deposition of alumina thin films still has its challenges. RF sputtering from compound targets can produce stoichiometric high-quality coatings. However, its notoriously low deposition rate makes it unsuitable for large-scale mass production. Dual magnetron sputtering (DMS), from the other hand, can provide industrially meaningful deposition rates from metallic targets and successfully to circumvent the problem with the disappearing anode. However, it requires a very fast and solid regulation, allowing for long-term stable operation at narrow operating range. Special attention needs to be paid for arc handling on the target and substrate and its influence on the regulating algorithm.

In this talk, the contemporary state of the art for industrial DMS deposition of aluminium oxide for tool and sensor application is presented. Different ways of regulating are discussed and compared. The properties of low- and high-temperature deposited alumina films are reported. Future technology improvements are also discussed.

11:40am **PP1-1-MoM-6 Control of Microstructure and Phase of Sputter-Deposited Tantalum Thin Films for Inkjet Device Applications***, **Brittney Burant [brittney.burant@hp.com]***, HP Inc, USA

Tantalum (Ta) has long been used in thermal inkjet devices and plays a crucial role in the ink ejection cycle. It forms the bubble nucleation surface that transfers heat from the resistor to the ink, and also acts as a cavitation barrier that mechanically and chemically protects the resistor from damage during bubble collapse. Ta has a bulk stable α-phase with BCC structure, however, the metastable β-phase has historically been used in inkjet devices. Microstructure and phase control of the thin film is important for promoting cohesive bubble nucleation during ink firing cycles and ensuring the device functions reliably through its lifetime.

When pursuing new product architectures with reduced die separation ratios, it was found that sputter-deposited Ta films began to exhibit mixed phases, compromising the integrity of the cavitation barrier. Much research has been done to characterize the phase selection of Ta, however not much is understood about the underlying mechanisms of β-phase initiation and many studies report contradictory process parameters for phase selection. Through our work, we were able to demonstrate that substrate pre-treatment was promoting mixed phase formation during deposition, independent of other deposition parameters. We further aim to show that both substrate roughening and native oxide of the passivation surface plays a key role in initiating β-phase film growth on the substrate, by modifying the pre-sputter etch conditions and characterizing the surface and subsequent Ta phase.

12:00pm **PP1-1-MoM-7 Dc Magnetron Sputtering Yield Amplification of C, Si, and Ge Doped with W, Cu, Ta, or Mo***, **Julio Cruz [juliocruz@ens.cnyn.unam.mx]***, Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México*; Rebecca Giffard,* Universidad de Guadalajara, Mexico*; Stephen Muhl, Marco Martínez,* Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México*; Roberto Sanginés, Roberto Machorro,* Centro de Nanociencias y Nanotecnología. Universidad Nacional Autónoma de México*; Efraín Chávez,* Instituto de Física. Universidad Nacional Autónoma de México

S. Berg in 1991 discovered the phenomenon called Sputtering Yield Amplification, SYA. The phenomenon is related to doping a sputtering target with atoms of different atomic mass than the target material. Such doping changes the collision cascade on the surface of the target, consequently increasing the number of ejected atoms from the target. In this work, we present a way of generating SYA of C and Si with two different types of co-sputtering experiments, in both by adding the doping element as small pieces on the surface racetrack. First, we increased the amount of W on C, Ge, and Si targets. Second, we increased the working gas pressure, i.e., the number of collisions in the gas phase, using a Si target and doping it with Cu, Mo, and, Ta. Then we studied the number of dopant atoms returning to the target racetrack surface and its spatial distribution. To determine their effect on the cascade of collisions and, consequently, the change in the target sputtering yield. With the Perfilometry and Rutherford Backscattering Spectrometry techniques, we measure the thickness of the deposited films and the total number of atoms deposited on the substrate. With Optical Emission Spectroscopy we analyze the intensity of the emission lines of neutral and ionized species in the sputtering plasma. Furthermore, with SIMTRA code we theoretically estimated the spatial distribution of atoms that were redeposited on the target. The results showed Si and C SYA doped with W. Somewhat similar results have been reported earlier. Furthermore, significant Si SYA doped with Cu, Mo, and Ta. These results may be interesting for materials that have both lower sputtering yield and important applications in the thin films industry.

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