New Horizons in Coatings and Thin Films
Room San Diego - Session F2-1

HiPIMS, Pulsed Plasmas and Energetic Deposition

Moderators: Tiberiu Minea, Université Paris-Sud, Jon Tomas Gudmundsson, University of Iceland

10:00am **F2-1-1 On Recycling in High Power Impulse Sputtering Magnetrons***, **Jon Tomas Gudmundsson**,* University of Iceland, Iceland*; N Brenning, M Raadu,* KTH-Royal Institute of Technology, Sweden*; T Petty, T Minea, D Lundin,* Université Paris–Sud, France

We will discuss a framework on how to quantify and understand large discharge currents in high power impulse magnetron sputtering (HiPIMS) discharges by investigating the role of self-sputter(SS-) recycling and working gas recycling. We find that above a critical current density $J_{crit} \approx 0.2$ A/cm^2, a combination of self-sputter recycling and working gas-recycling is the general case. The relative contributions of these recycling mechanisms, in turn, influence both the electron energy distribution and the stability of the discharge [1]. For high self-sputtering yields, above $Y_{SS} \approx 1$, the discharges become dominated by SS-recycling, contain only a few hot secondary electrons from sheath energization, and have a relatively low effective electron temperature. Here, stable plateau values of the discharge current develop during long pulses, and these values increase monotonically with the applied voltage. For low self-sputtering yields, below $Y_{SS} \approx 0.2$, the discharges operated above J_{crit} are dominated by working gas recycling, have a significant sheath energization of secondary electrons and a higher effective electron temperature, and the current evolution is generally less stable. For intermediate values of Y_{SS} the discharge character gradually shifts between these two types. In addition, these new insights on ion recycling have been applied to a series of selected sputter materials. For high currents a discharge with Al target develops almost pure self-sputter recycling, while a discharge with Ti target exhibits close to a 50/50 combination of self-sputter recycling and working gas-recycling [2]. However, if the Ti target is operated in a reactive Ar/O_2 gas mixture, it is found that working gas-recycling is dominating [1] and that the Ar^+-ions contribute almost solely to the discharge current [3].

[1] N. Brenning et al. Plasma Sources Sci. Technol. (submitted 2017)

[2] C. Huo et al., J. Phys. D: Appl. Phys. 50 354003 (2017).

[3] J. T. Gudmundsson et al. Plasma Sources Sci. Technol. 25, 065004 (2016).

10:20am **F2-1-2 Electron Density at the Sheath Edge of a HiPIMS Plasma***, A Hecimovic, **Julian Held**, V Schulz-von der Gathen, W Breilmann, C Maszl, A von Keudell,* Ruhr-Universität Bochum, Germany

In High power impulse magnetron sputtering (HiPIMS) a magnetron discharge is operated with short, high-voltage pulses with a length in the order of 100 µs at power densities of several kW/cm^2, creating a highly dense plasma. At high discharge currents, the plasma is not homogeneous but is instead organized into distinct zones of high plasma emission which rotate in ExB direction with velocities of about 10 km/s. The strong emission indicates an elevated electron density inside those so called "spokes". Up to now, no measurement of the electron density inside the spokes has been performed. In this contribution, small inserts in the target surface were used to probe the local current density. Simple sheath theory was then applied to derive the electron density at the sheath edge. The electron density was found to be a few 10^{20} m^{-3}. The electron density inside the spokes was about 25% higher than in the plasma between the spokes.

10:40am **F2-1-3 Spatially Resolved Investigation of Transport and Redeposition Processes during HiPIMS by Means of Optical Diagnostics and In-vacuum XPS Analysis of Magnetron Targets***, **Sascha Monje**, V Layes, A von Keudell,* Ruhr-University Bochum, Germany*; T de los Arcos,* University Paderborn, Germany*; V Schulz-von der Gathen, C Corbella,* Ruhr-University Bochum, Germany

The distribution of redeposited species and their oxidation states were evaluated with in-vacuum X-ray photoelectron spectroscopy (XPS) of magnetron targets after reactive and non-reactive high power impulse magnetron sputtering (HiPIMS). The investigation was performed for various metal targets (all circular with 50 mm diameter). In addition to regular targets, 'composite' targets were used which are made of a regular metal target, where a second cylindrical shaped metal insert is located in the racetrack center. The insert of these targets acts as a marker for the species transport in the plasma.

The HiPiMS discharge was used at several power conditions and was characterized with a fast imaging CCD-camera as well as optical emission spectroscopy (OES). The target surface composition was evaluated with XPS after an in-vacuo transfer to an UHV chamber.

With the characterization of the plasma on one hand and the redistribution of material on the other hand, it was possible to connect the transport and the discharge behavior. It was found that the lateral transport and redeposition of species are influenced by the appearance of spokes. Furthermore, a correlation between oxidation state and the local surface condition has been found.

11:00am **F2-1-4 Time-resolved Ion Energy and Charge Distributions in Pulsed Cathodic Arc Plasmas of Nb-Al Cathodes in High Vacuum.***, **Siegfried Zoehrer**,* Montanuniversität Leoben, Austria*; A Anders,* Lawrence Berkeley National Laboratory, USA, and now at Leibniz Institute of Surface Engineering (IOM), Germany*; R Franz,* Montanuniversität Leoben, Austria

Cathodic arcs are utilized in industry for a wide variety of applications. For instance, to synthesize functional thin films and coatings, to form energetic metal ion beams for ion implantation, or in high current switches. Although there has been tremendous progress in the last decades, the physics responsible for the observed plasma properties are still a matter of dispute. That is particularly the case for multi-element cathodes, which can play an essential role in all given examples. An often overlooked criterion, especially in DC arc plasma analysis, is the typically occurring neutral background of metal atoms in cathodic arcs. It perturbs initial ion energy and charge distributions, which makes it difficult to get information of the near-cathode plasma and also to relate effects to the cathode composition. Therefore, we use a recently developed method to record time-resolved ion charge state and energy distributions in pulsed vacuum arc plasmas from Nb-Al cathodes. This model system consists out of three different Nb-Al compositions, as well as pure Nb and Al cathodes. The results visualize ion detections of 600 µs plasma pulses, extracted 0.27 m from the cathode resolved in energy and time. They show a heavy influence of neutrals on ion charge state fractions and, to a lesser extent, on ion velocity distributions, which can be observed in the time evolution of these properties. Subsequently, fundamental hypotheses applying to multi-element cathodes, like the „velocity rule“ or the „cohesive energy rule“ are tested on their applicability to early and late stages of the pulse. The results also show a strong material dependency, which appears for initial as well as saturated ion charge states and velocities. That leads to a quite different behavior for single-element cathodes and Nb-rich/Al-rich multi-element cathodes, where the latter generally show lower velocities and less multiply charged Nb ions. Apart from their fundamental character, these findings can be useful for optimizing or designing plasma properties for applications by actively utilizing effects on ion distributions caused by multi-element cathode materials and charge exchange with neutrals.

11:20am **F2-1-5 Investigations on the Substrate Bias Influence on Reactive High Performance Plasmas***, K Bobzin, T Brögelmann, N Kruppe, **Martin Engels**,* Surface Engineering Institute - RWTH Aachen University, Germany

High performance plasma processes are subject of several studies. For the high power pulse magnetron sputtering (HPPMS) and hybrid processes composed of HPPMS and direct current magnetron sputtering (dcMS), respectively, investigations regarding correlations between plasma and coating properties and the process parameters have been carried out. These investigations especially focus on the HPPMS pulse parameters frequency f and pulse-on-time t_{on}, the power density at the target and the process gas pressure. However, another important aspect with respect to industrial coating processes is the substrate bias, which is used to accelerate ionized coating forming species to the substrate. This acceleration results in dense and homogeneous coatings due to subplantation of these species into the coating. In order to understand this mechanism, the correlation of substrate-sided plasma properties and the resulting coating properties is a reasonable approach. Nevertheless, it is complicated to implement substrate-sided plasma diagnostics which are closely adapted to industrial coating processes using substrate bias. Hence, the present work focuses on strategies to conduct quantifiable investigations on the high performance plasma properties using substrate bias. These investigations were conducted for reactive HPPMS and dcMS/HPPMS (Cr,Al)N and (Cr,Al)ON processes. The coating systems were chosen, since they are widely used as protective coatings for many tool applications. The strategies were developed for the plasma diagnostics optical emission spectroscopy, Langmuir probe and energy resolved mass spectroscopy in an industrial scale coating unit. A varying substrate bias with values from $U_B = 0$ V to $U_B = -250$ V was used to validate the developed methodology. The results on the substrate-sided plasma properties like the chemical composition, ionization or Debye sheath thickness were correlated with (Cr,Al)N and (Cr,Al)ON coating properties, i.e. the morphology by scanning electron microscopy, the chemical composition by means of glow discharge optical emission spectroscopy, residual stress by means of cantilever sensor chips as well as the universal hardness and the indentation modulus by nanoindentation. The correlation was conducted using an artificial neural network (ANN). With the developed methodology it was possible to identify significantly changing plasma properties at the substrate side when varying the substrate bias. The correlating coatings properties and the output data of the ANN were used to validate the methodology for the analysis of industrial coating processes.

11:40am **F2-1-6 The Impact of a Positive Pulse in HIPIMS Films***, **Jason Hrebik,*** Kurt J. Lesker Company, USA

HIPIMS technology is on the rise and starting to be qualified in more and more applications in the thin film coating industry. Low power HIPIMS options have enabled more R&D with the technology and as a result, new ways of utilizing and optimizing the technology has been found and published.

Test data will be presented showing various performance advantages of applying a positive pulse (reversing the voltage on the target) during HIPIMS applications. During HIPIMS applications a negative pulse at extremely high densities is applied to the target creating a highly ionized plasma at the target surface. Applying a positive pulse prior to the negative pulse extinguishing extends the plasma and repels metal ions that have not recirculated yet toward the substrate. As a result, higher deposition rates, higher ion fraction, denser films, and less film stress is achieved. In addition, the positive pulse option has a significant effect on reactive coating application in that the positive pulse neutralizes the target resulting in fewer arcs, minimizing the disappearing anode effect.

Data will show that the increase in sputtering rate as a result is approximately 15%. Increasing voltage also has a positive impact on the rate. In addition, the higher density and reduced film stress will be shown in provided performance data.

Parameters can be adjusted to optimize performance for various process requirements such as Height of Pulse, Width of Pulse, and Delay of Pulse. This is a key feature of the technology due to the fact that there are variable requirements based on the application that may need to be tuned and altered. Examples of how these changes can alter performance will be presented.

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