Advanced Surface Engineering
Room: C4 - Session SE2-MoM

Pulsed Plasmas in Surface Engineering
Moderator: J. Patscheider, EMPA, Switzerland

8:20am **SE2-MoM1 Industrialization of Metal Ion Sputtering**, ***R. Cremer***, KCS Europe **INVITED**

Since its introduction by Kouznetsov et al. in 1999, the HIPIMS technology has seen a remarkable rise in interest from both academic and industrial viewpoint. Although the high ionization of the plasma and the resulting advantages for industrial sputter applications have been verified more than a decade ago, industrial usage of the metal ion sputtering technology has been limited due to various technical drawbacks.

Only recently, a various number of authors have reported the overcome of the hitherto existing disadvantages of the technology like low deposition rate, biasing issues, arcing and reliability of the technology.

This paper gives an overview on the industrialization of metal ion sputtering in various applications. Special focus will be given to the comparison of ionization in different coating technologies like sputtering, metal ion sputtering, arc ion plating and thermionic arc evaporation. The paper will also comment on future options and limitations of industrial metal ion sputtering.

9:00am **SE2-MoM3 Structural and Mechanical Behavior of Fullerene-Like and Amorphous Carbon Nitride Thin Films Deposited by HPPMS**, ***S. Schmidt***, *G. Greczynski*, Linköping University, Sweden, *E. Broitman*, Carnegie Mellon University, *L. Hultman*, Linköping University, Sweden

The structural and mechanical properties of fullerene-like (FL) and amorphous carbon nitride (CN_x) films were deposited using High power pulsed magnetron sputtering (HPPMS) in an industrial CC-800/9 CemeCon chamber and compared with films deposited by DC magnetron sputtering mode of operation.

Films of 1 µm and 2 µm thickness were grown on Si and steel substrates, respectively. Carbon nitride films were deposited via HPPMS from a high purity graphite target in an Ar/N_2 discharge at 400 mPa, the N_2 fraction varied from 0 to 0.5 and different substrate temperatures ranging from ambient temperature to 300°C were chosen. Furthermore, a novel HPPMS substrate pretreatment employing two HPPMS power supplies was used to optimize the adhesion of the films: the first power supply established the discharge; the second produced a pulsed substrate bias. The created Cr-plasma cleaned the substrate surface and formed a Cr-containing gradual interface into the substrate. X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM), and transmission electron microscopy (TEM) were used to study the microstructure of both, the films and the interfaces. The hardness and the elastic recovery of the CN_x films were measured using nanoindentation. A deposition process window is demonstrated for the growth of dense fullerene-like (FL) film structures consisting of curved, frequently intersecting, and highly in-plane oriented basal planes.

9:20am **SE2-MoM4 Effects of the Working Pressure and Pulse Parameters on the Ion Energy and Mass Distributions in Modulated Pulse Power Sputtering Plasma**, ***W.D. Sproul***, Reactive Sputtering, Inc., *J. Lin, J.J. Moore, B. Mishra*, Colorado School of Mines, *J.A. Rees*, Hiden Analytical Ltd, UK, *Z.L. Wu, J. Wang*, Colorado School of Mines, *R. Chistyakov, B. Abraham*, Zond/Zpulser

The modulated pulse power (MPP) sputtering technique is a variation of high power pulsed magnetron sputtering (HPPMS) that generates a high ionization density plasma by manipulating the pulse shape, intensity, and duration. In this study, the time-averaged ion energy and ion mass distributions of the MPP plasma generated during sputtering a metal Cr target in pure Ar at different working pressures, pulse frequencies, and the strong ionization periods were investigated. The MPP plasma was studied using an electrostatic quadrupole plasma mass spectrometer which was installed parallel to the target surface in a closed field unbalanced magnetron sputtering system. It was found that an increase in the working pressure led to an increase in the peak ion flux and a decrease in the high ion energy tail. An increase in both the gas and metal ion species was observed as the pulse frequency was increased using the same pulse shape. Additionally, the effects of different combinations of the weak and strong ionization durations on the ion energy distributions of the gas and metal ion species will be reported.

10:00am **SE2-MoM6 Effects of HiPIMS Plasma Transport on Thin Film Deposition**, ***D. Lundin***, Linköping University, Sweden, *N. Brenning, M.A. Raadu*, Royal Institute of Technology, Sweden, *U. Helmersson*, Linköping University, Sweden

A new exciting development of magnetron sputtering was achieved when introducing high power impulse magnetron sputtering (HiPIMS). HiPIMS is one of the most promising improvements of common IPVD techniques and is already making its way to industrial applications. The HiPIMS plasma generates large quantities of ions of the sputtered material due to a high plasma density, but also acceleration of the ions increasing the bombardment of the growing film without using a substrate bias voltage. Also observed is a lower deposition rate for HiPIMS than that obtained for conventional DC sputtering, using the same average power. In order to optimize the process, controlling ion acceleration and increasing deposition rate, the mechanisms for transport of charged particles in this type of plasma need to be known. In the present work, it is shown that the electron mobility across the magnetic field is enhanced by typically an order of magnitude during the HiPIMS discharge compared to DC magnetron sputtering. This cannot be explained by classical theory of diffusion and electrical conductivity or Bohm diffusion. The transport is directly reflected by an anomalously low azimuthal-to-discharge current ratio, $J_\varphi / J_D = 2$. On the microscopic scale, the anomalous transport can be shown to be mediated by observed azimuthal electric field oscillations in the lower hybrid range. Furthermore, new insights from experimental data and plasma discharge modeling will be presented, which show that a large fraction of the ionized species are attracted back towards the target, either by electric fields in the bulk of the plasma, or by the stronger local fields in the cathode sheath. In this context, it is demonstrated that the effect of the anomalously high electron mobility to reduce the bulk **E** field is important to understand and control. The study also verifies that the resistive friction force, $\mathbf{F}_{i,\varphi}$, associated with the anomalous resistivity, can accelerate the ions azimuthally, as is shown both indirectly from changes in the deposition patterns, and directly by mass spectrometry.

10:40am **SE2-MoM8 Deposition Rates of High Power Impulse Magnetron Sputtering: Physics and Economics**, ***A. Anders***, Lawrence Berkeley National Laboratory **INVITED**

Deposition by high power impulse magnetron sputtering (HIPIMS) is considered by some as the new paradigm of advanced sputtering technology, yet this is met with skepticism by others for the reported lower deposition rates, if compared to direct current (DC) sputtering of equal average power. In this contribution, absolute and relative (normalized) deposition rates are compared, and the underlying physical reasons for differences are discussed, including (i) ion return for self-sputtering, (ii) the less-than-linear increase of the sputtering yield with increasing ion energy, (iii) yield changes due to the shift of species responsible for sputtering, (iv) change in plasma impedance and sheath voltage, (v) changes in film density, (vi) noticeable losses in the switch module, (vii) changes of the magnetic balance and particle confinement of the magnetron due to self-fields at high current, and (viii) superposition of sputtering and evaporation for selected materials. The situation is even more complicated in reactive systems where the target surface chemistry is a function of the discharge conditions. While generally these factors imply a reduction of the normalized deposition rate, increased rates have been reported for certain conditions. Finally, some points of economics and “value added” are considered.

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11:20am **SE2-MoM10 Optical Diagnostics of HIPIMS Discharges: Dependence of Film Growth on Control Parameters**, ***M. Lange***, UTC and AFRL/RXBT, *J. Jones*, AFRL/RXBT, *C. Muratore*, UTC and AFRL/RXBT, *A. Reed*, AFRL/RXBT, *A. Waite*, UTC and AFRL/RXBT, *A. Voevodin*, AFRL/RXBT

High power impulse magnetron sputtering is a physical vapor deposition process distinguished by its capability to produce a high flux of ionized target material incident upon growing film surfaces. This characteristic gives the process advantages over conventional dc sputtering in that the orientation and relative density of thin films can be controlled by modulating the energy of ions constituting the film material. Unfortunately, the deposition rate in HIPIMS is often lower than in standard magnetron sputtering when processes conducted with the same time-averaged power are compared. The deposition rates for HIPIMS processes are dependent upon the power waveforms to the target, as well as the ionization energy, self sputtering rate, atomic mass, and other physical properties of the sputter target material. Correlation of target materials with thoughtfully selected properties to resultant plasma characteristics can reveal the nature of these relationships. For example, the ionization energies of hafnium and titanium are similar (6.8 eV), but their atomic masses of 178 amu and 48 amu respectively, affect the deposition rate in addition to the temporal-spatial plasma distributions, which were measured here using optical and electrostatic diagnostics. Studies of these materials provide insight on the effect of target mass on ion transport and film growth rates. Substrate bias and pulse duration have also been shown to effect the optical emissions from the plasma generated during HIPIMS operation. Correlation of these plasma characteristics to the structure and properties of elemental and compound thin films will be presented.

11:40am **SE2-MoM11 Time-resolved Plasma Characterization in Modulated Pulse Power (MPP) Magnetron Sputtering**, ***A.N. Cloud***, *R.E. Flauta, M.J. Neumann, S.L. Rohde, D.N. Ruzic*, University of Illinois at Urbana-Champaign

High power impulse magnetron sputtering (HIPIMS/HPPMS) has attracted considerable attention from industry due its ability to produce thin films and features of excellent adhesion, superior density, decreased roughness, and extreme conformity. The intense pulsed plasma density – on the order of 10^{18} m^{-3} – provides a large concentration of metal ions that can be used to produce high-quality, homogeneous coatings. The high ionization fraction at the substrate allows for fine control of the sputtered species during deposition.

Modulated pulse power (MPP) can be employed to shape an arbitrary voltage waveform that is applied to the cathode. This programming freedom allows control over pulse duration, intensity, duty cycle, and average power. Voltage oscillations during the 1.0 – 3.0 ms pulse on the order of 25-65 kHz induce instabilities in the plasma discharge that may have a marked effect on the level of ionization within the discharge and distribution of the metal ions. The oscillation frequency range corresponds to the expected ion cyclotron angular frequencies. Past investigations of MPP have only revealed time-averaged plasma parameters, but knowledge of events during the pulse is required to further understanding of the physical mechanisms involved.

MPP discharges produced with a 1000 cm^2 circular planar magnetron were characterized. A gridded energy analyzer and quartz crystal microbalance were used to measure a higher ionization fraction than with conventional magnetron sputtering under a variety of deposition conditions. Nominal values of approximately 6% were attained for the sputtering of titanium at power densities as low as 100 W/cm^2. The energy spectrum and flux of these ions at the substrate location were also measured, finding the incident metal ions to be of low energy between 1 and 4 eV. Time-resolved plasma properties including saturation current, electron temperature, and density are measured and mapped over the three-dimensional space between the sputter target and substrate using a triple Langmuir probe. Plasma density is shown to decrease by greater than an order of magnitude between pulses. The effects of pulse duration, current density, pulse shape, switching frequency, and target material on the discharge are explored and discussed.

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