Advanced Surface Engineering
Room: 204 - Session SE+PS-FrM

Pulsed Plasmas in Surface Engineering
Moderator: A. Erdemir, Argonne National Laboratory

8:20am **SE+PS-FrM1 On the Plasma Parameters in the High Power Impulse Magnetron Sputtering Discharge (HiPIMS)**, ***J.T. Gudmundsson***, University of Iceland **INVITED**

The development of ionized physical vapor deposition (IPVD) was mainly driven by the formation of metal and nitride thin films into deep, narrow trenches and vias that are essential in modern microelectronics. More recently, the control of the ion energy and direction of the deposition species has proved to be an important physical tool in the growth process of new materials and new structures. Over the past few years, various ionized sputtering techniques have appeared that show a high degree of ionization of the sputtered atoms, in the range 50 - 90 %. This is often achieved by the application of a secondary discharge to a magnetron sputtering discharge, either inductively coupled plasma source (ICP-MS) or a microwave amplified magnetron sputtering[1]. High power impulse magnetron sputtering (HiPIMS) is a more recent sputtering technique that utilizes ionized physical vapor deposition (IPVD)[1,2]. High density plasma is created by applying a high power pulse to a planar magnetron discharge. Measurements of the temporal and spatial behavior of the plasma parameters indicate peak electron density of the order of 10^{19} m^{-3}, that expands from the target with a fixed velocity that depends on the gas pressure[3]. The high electron density results in a high degree of ionization of the deposition material. Fractional ionization of the sputtered material has been measured to be over 90 %[2]. The ions are controllable with respect to energy and direction as they arrive to the growth surface. The spatial and temporal variation of the plasma parameters, electron density, electron energy, plasma potential and ion energy, in a HiPIMS discharge are reviewed. The plasma physics of the HiPIMS will be discussed as well as some of applications of the HiPIMS technique.

[1]U. Helmersson, M. Latteman, J. Bohlmark, A. P. Ehiasarian, and J. T. Gudmundsson, Ionized Physical Vapor Deposition (IPVD): A Review of Technology and Applications, Thin Solid Films 513 (2006) 1-24

[2]U. Helmersson, M. Lattemann, J. Alami, J. Bohlmark, A.P. Ehiasarian, and J.T. Gudmundsson, Proceedings of the 48th Annual Technical Conference of the Society of Vacuum Coaters, April 23-28, 2005, Denver, CO, USA, p.458

[3] J.T. Gudmundsson, J. Alami, and U. Helmersson, Spatial and temporal behavior of the plasma parameters in a pulsed magnetron discharge, Surf. Coat. Technol. 161 (2002) 249 .

9:00am **SE+PS-FrM3 Deposition of Metal Oxide Coatings using Reactive High Power Impulse Magnetron Sputtering**, ***E. Wallin***, *M. Aiempanakit,* Linköping University, Sweden, *T.I. Selinder, E. Coronel,* Sandvik Tooling, Sweden, *U. Helmersson*, Linköping University, Sweden

Metal oxides have been deposited using reactive high power impulse magnetron sputtering (HiPIMS) of metal targets in Ar/O_2 gas mixtures. The use of HiPIMS has in previous studies of deposition of alumina been shown to drastically influence the process characteristics compared to conventional reactive sputtering [Wallin and Helmersson, Thin Solid Films, in press]. Under suitable conditions, oxide formation on the target was found to be suppressed, and the hysteresis effect commonly observed as the gas flow is varied during conventional sputtering was reduced, or even completely eliminated, using HiPIMS. In the present work, these investigations are extended to a wider range of process parameters as well as to other material systems, including CeO_2, in order to better understand the reactive process. Based on this, reasons for the altered process characteristics will be discussed. Moreover, film properties of alumina deposited by this type of process have been investigated. α-alumina was found to form readily on both cemented carbide and Mo substrates at a temperature as low as 650 °C. α phase growth was retained over the studied range of substrate bias voltages (from floating potential to -100 V), while growth at lower temperatures resulted in the formation of γ-alumina at 575 °C and x-ray amorphous films at 500 °C or lower. The film microstructure was studied using electron microscopy techniques, revealing a plate-like structure of the α-alumina films with wider grains and a denser structure for higher bias values. Reasons for the phase composition and microstructure observed with different process parameters will be discussed together with possible pathways for further reduction of the α-alumina growth temperature and improvements of the microstructure.

9:20am **SE+PS-FrM4 A Mass/Energy Analysis of the Plasma during Modulated Pulse Power Sputtering**, ***W.D. Sproul***, Reactive Sputtering, Inc., *J. Lin, J.J. Moore, M. Hasheminiasari,* Colorado School of Mines, *R. Chistyakov, B. Abraham*, Zond, Inc./Zpulser, LLC

During modulated pulse power (MPP) sputtering, there are multiple steps within the overall pulse. Usually there are 3 steps, but there can be many more if needed. The first step is the application of a high voltage to the cathode that ignites a weakly ionized sputtering plasma. This weakly ionized plasma is allowed to stabilize in step 2, and then the voltage to the cathode is increase to transition the plasma into a strongly ionized plasma in step 3. This strongly ionized plasma is characterized by a significant increase in the current to the cathode accompanied with a moderate voltage increase. The overall power to the cathode is thus also greatly increased. At the substrate when a bias is used, there is also an increase in the substrate ion current density during step 3, and this ion current density increases as a function of the peak power. The deposition rate for the Cr films is a function of the peak power on the target, but there is a pronounced increase in the deposition rate when the peak power exceeds approximately 100 kW. In this study, a mass/energy analyzer was used to characterize the species in the plasma during the different steps of MPP sputtering of Cr films. Cr plus one ions were readily detected by the mass/energy analyzer in step 3 of the pulse, but it was more difficult to detect multiply charged Cr ions due to the location of the analyzer with respect to plasma and the target. It is possible that multiply ionized Cr ions are not detected due to charge exchange collisions in the plasma. The changes in the species in the plasma will be correlated with observed changes in the structure and properties of the Cr films deposited under different peak power conditions.

9:40am **SE+PS-FrM5 Process, Structure and Properties of Chromium and Chromium Nitride Coatings Synthesized using Modulated Pulse Power (MPP) Sputtering**, *J. Lin, Z. Wu,* Colorado School of Mines, *W.D. Sproul,* Reactive Sputtering, Inc., *B. Mishra,* ***J.J. Moore***, *M. Hasheminiasari,* Colorado School of Mines, *R. Chistyakov, B. Abraham*, Zond, Inc./Zpulser, LLC

Modulated pulse power (MPP) sputtering is a variation of high power pulse magnetron sputtering that overcomes the rate loss issue and achieves the enhanced plasma ionization through modulation of the pulse shape, intensity, and duration. In the current studies, Cr and CrN coatings were synthesized using MPP under different pulse durations and different combinations of the voltage rise and fall times, which were found to exhibit strong influence on the deposition parameters. It was found that the target power, voltage, current, and ion current density were increased with an increase in the long pulse durations and the voltage rise time. For Cr coating depositions, the MPP exhibits higher deposition rates than in the dc conditions when the average power is above 10-12 W/cm^2. A high deposition rate of 230 nm/min for the Cr coating deposition can be achieved with optimized pulsing parameters. The structure of the Cr and CrN coatings were characterized using x-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The mechanical and tribological properties were measured by performing depth-sensing nanoindentation tests, micro-scratch tests and a ball-on-disc wear test in ambient atmosphere. It was found that the microstructure of the coatings changed from large columnar grains to dense and fine nano grains with an increase in the power and ion current densities on the target. A high hardness of 18 GPa has been achieved in Cr coatings deposited with an average power density of 21 W/cm^2 and an ion current density of 1.2 A/cm^2 on the target with a floating substrate bias.

10:00am **SE+PS-FrM6 Deposition Rate of High-Power-Pulse Magnetron Sputtering Processes**, ***J. Emmerlich***, *S. Mráz, S. Konstantinidis,* RWTH Aachen University, Germany, *R. Snyders,* University of Mons, Belgium, *J.M. Schneider*, RWTH Aachen University, Germany **INVITED**

In high power pulsed magnetron sputtering (HPPMS), a large power density is applied giving rise to a high degree of ionization. From an application point of view, the major drawback of this technology is the considerably lower metal deposition rate as compared to DC magnetron sputtering. Using transport-of-ions-in-matter simulations (TRIM), it is shown that the apparently low deposition rate can be understood based on the non-linear energy dependence of the sputtering yields. The simulations are consistent with deposition-rate measurements on Cu films as well as with published deposition rate data for Ti [Konstantinidis et al., J. Appl. Phys. 99, 013307 (2006)]. TRIM simulations in combination with deposition rate experiments as a function of pulse width using Cu, W, and Ti as target materials reveal predominantly self-sputtering during Cu depositions. For W as well as Ti discharges, Ar contributes significantly more to sputtering, which may be explained by the low metal-self-sputtering yield. HPPMS deposition rates during reactive sputtering are reported to be comparable or even larger compared to DC magnetron sputtering rates [Wallin and Helmersson, Thin Solid Films in press]. Target erosion rate measurements for an HPPMS discharge exhibit two orders of magnitude larger erosion rates compared to DC magnetron sputtering.

10:40am **SE+PS-FrM8 Effects on Thin Film Growth Due to Anomalous Transport in High Power Impulse Magnetron Sputtering**, ***D. Lundin****, P. Larsson, E. Wallin,* Linköping University, Sweden*, M. Lattemann,* TU Darmstadt and Forschungszentrum Karlsruhe GmbH, Germany*, N. Brenning,* Royal Institute of Technology, Sweden*, U. Helmersson*, Linköping University, Sweden

In this study, the effect of a previously reported anomalous transport[1] on thin film growth in high power impulse magnetron sputtering (HiPIMS) has been investigated for the case of a planar circular magnetron. It was found that a large fraction of ions are transported radially outwards in the vicinity of the cathode, across the magnetic field lines, leading to enhanced deposition rates directly at the side of the cathode (on a substrate oriented perpendicular to the target surface). An important consequence of this type of mass transport parallel to the target surface is that the fraction of sputtered material reaching a substrate placed directly in front of the target is substantially lower in HiPIMS compared to conventional direct current magnetron sputtering (dcMS). This would help to explain the lower deposition rates generally observed for HiPIMS compared to dcMS. Moreover, time-averaged mass spectrometry measurements of the energy distribution of the cross-field transported ions were carried out. The measured distributions show a direction-dependent high-energy tail, which can be explained by an increase in the azimuthal force on the ions, exerting a volume force on the ions tangentially outwards from the circular race track region. These results are in agreement with predictions as well as recent modeling results of the anomalous transport mechanism.

[1] D. Lundin, U. Helmersson, S. Kirkpatrick, S. Rohde, and N. Brenning, Plasma Sources Sci. Technol. 17, 025007 (2008).

11:00am **SE+PS-FrM9 High Power Impulse Magnetron Sputtering of Ti-Si-C Multifunctional Thin Films**, ***M. Samuelsson***, Linköping University, Sweden*, H. Högberg, H. Ljungcrantz,* Impact Coatings, Sweden*, U. Helmersson*, Linköping University, Sweden

Nanocomposite Ti-Si-C thin films grown by dc-magnetron sputtering (dcMS) are interesting for many applications, such as in electrical contacts. This is due to a property envelope including low contact resistance, ductility and hardness that can be combined. Other areas of applications are also suggested, which call for increased possibility to design the material for specific needs. A promising deposition technique is high power impulse magnetron sputtering (HiPIMS), which offers a high degree of ionization of the sputtered material not found in conventional dc-magnetron sputtering. Growth from ions instead of neutrals is likely to further increase the possibility of designing the film microstructure and thereby the properties. In this study we have investigated sputtering from a Ti_3SiC_2 target by HiPIMS and compared the technique with dcMS. The techniques have been compared for different process pressures and substrate bias voltages using a pilot plant deposition system under production like conditions. The results show that the obtained HiPIMS growth rate was approximately 13% of that of dcMS for comparable average powers. Further studies employing SEM, TEM, XRD measurements, surface resistivity and film adhesion will be presented.

11:20am **SE+PS-FrM10 Modulated Pulse Power Deposition of Aluminum Oxide Nanometer Scale Multilayer Films**, ***R. Chistyakov***, Zond Inc.*, B. Abraham,* Zpulser LLC*, W.D. Sproul,* Reactive Sputtering, Inc.*, J.J. Moore, J. Lin*, Colorado School of Mines

Modulated pulse power (MPP) sputtering is a versatile high power pulse magnetron sputtering technique in which there can be multiple voltage steps within a pulse. Different levels of applied voltage in the same voltage pulse will generate different power levels for the magnetron discharge. Usually each pulse shape has a weakly ionized plasma (low power magnetron discharge) step that was generated first, and then the second stage that has a strongly ionized plasma (high power magnetron discharge) by applying a voltage increase to the cathode. These arbitrary voltage pulse shapes can be used within a given deposition run to form multilayer film structure. Therefore every layer can be sputtered with a different voltage pulse shape. In this study, two different voltage pulse shapes were selected. The first pulse had a shorter duration that the second pulse, but by varying the repetition rate the same average power could be delivered during the sputtering of each layer. The peak power applied to the plasma was greater during the second pulse, which meant that a greater amount of energy was applied to the process during the peak power phase of the second pulse. The difference in the applied energy between the two MPP pulse shapes was used during the reactive sputter deposit of aluminum oxide films . This two-pulse approach did produce a nanometer scale layering of the aluminum oxide coatings, which was observed in a scanning electron microscope. The thickness and structure of each nanometer scale layer was controlled by varying the output voltage pulse shape of the MPP plasma generator and deposition time. The layering of the aluminum oxide affected not only the structure of the films, but it also affected the mechanical properties of the films. The film structure, orientation, and mechanical properties were analyzed and measured, and the results of the film property measurements will be presented.

11:40am **SE+PS-FrM11 The Specification and Optimization of HIPIMS Power Supply Parameters**, *D. Ochs,* Huettinger Electronics GmbH, Germany*, P. Ozimek,* Huettinger Electronics Sp. z.o.o., Poland*, **A.G. Spencer***, Alacritas Consultancy Ltd., UK

HIPIMS is a rapidly emerging technique for surface modification. It is well know the improvements in surface properties that can be acheived (in particular in mechanical properties). What is less well known is how to specify and operate the HIPIMS power supply. There are many aspects of the HIPIMS power supply that need to be specified at time of purchase, and adjusted for process optimization (average power, pulse power, pulse frequency, pulse length). Usually HIPIMS users are upgrading from a sputtering or evaporation process. These new HIPIMS parameters are therefore unfamiliar. This paper details the effects of each of these parameters, gives examples, and guidance on specifying a HIPIMS power supply.

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