### **Tuesday Morning, October 30, 2012**

Advanced Surface Engineering Room: 22 - Session SE+PS-TuM

#### **Pulsed Plasmas in Surface Engineering**

Moderator: C. Mitterer, University of Leoben, Austria

#### 8:00am SE+PS-TuM1 Strong Localization of Ionization in High Power Impulse Magnetron Sputtering in Reactive and Non-Reactive Gas Environments, A. Anders, Lawrence Berkeley National Laboratory INVITED

Self-organized structures of plasmas in high power impulse magnetron sputtering (HiPIMS) have recently been observed by several groups. The structures move along the racetrack in the ExB drift direction but only with about 10% of the electron drift velocity (E and B are the electric and magnetic field vectors). The bright structures are zones of concentrated ionization: they can be related to the amplification mechanism of selfsputtering and gas recycling. The ionization zones can readily be observed with fast cameras providing time resolution of 1 microsecond or better. The situation will be illustrated with images taken with a gated intensified frame image camera (Princeton Instruments) and with an intensified streak camera (Hamamatsu). Both end-on and side-on views to the magnetron are utilized. The side-on view reveals that each dense ionization zone is associated with an electron beam leaving the target region. Such beam maintains the quasineutrality of the plasma as ions are "evacuated" towards the target. The motion of the ionization zones is not a motion of plasma but a displacement of the region of greatest ionization. Its speed is mainly limited by the inertia of ions. The mechanism will be discussed and supported with images taken for various targets in noble and reactive gas atmospheres. Finally, the consequences of the localization of ionization for film formation will be considered in the greater context of energetic condensation.

#### 8:40am SE+PS-TuM3 The Magnetic Field Configuration's Effect on Plasma Parameters in High-Power Pulsed Magnetron Sputtering, H. Yu, L. Meng, P. Raman, T.S. Cho, D.N. Ruzic, University of Illinois at Urbana Champaign

Magnetic field design is crucial in DC magnetron sputtering operation, but has been largely overlooked in high power pulsed magnetron sputtering (HPPMS). In a HPPMS discharge, plasma disperses after each short pulse, unlike in the DC operation which requires a good magnetic confinement to maintain the plasma. It is thus interesting to study the effect of magnetic field configuration on HPPMS discharge for further optimization. A special magnet pack was fabricated with the magnet positions fully adjustable. Different designs were made, with the corresponding magnetic field calculated by Comsol. For example, the magnetic field (B) strength in the racetrack was varied, as 200, 500, and 800 Gauss. Different racetrack shapes were used, such as ring-like and bean-like. Designs were also made to have incomplete racetrack, and weak racetrack in which piles of magnets were placed evenly across the whole pack and polarities of adjacent piles were opposite. These magnetic configurations were then tested in a large magnetron system with a 36 cm target using a conventional HPPMS plasma generator. The I-V discharge characteristics were measured. A timeresolved triple Langmuir probe was employed to study the temporal evolution of electron temperature (Te) and density (ne) across the substrate. Ionization fractions of sputtered metal were also measured using quartz crystal microbalance combined with electrostatic filters. The results showed that higher B field strength and longer racetrack produced higher pulse current. The 500 Gauss configuration however had higher ne in the pulse than 800 Gauss did, likely because it allowed easier plasma diffusion. In all the designs with a racetrack, the distribution of ne on the substrate was nonuniform that n<sub>e</sub> was typically about 10 times higher right below the racetrack than at the center. The weak racetrack configuration was found to work and showed certain superiority over the normal DC magnet design. The plasma was generated from almost the entire target surface indicating an improvement in target utilization without the need for rotation. The discharge current was comparable with the racetrack designs. Furthermore, the plasma density distribution over the substrate was very uniform.

#### 9:00am SE+PS-TuM4 Plasma Generation and Transport in High-Power Pulsed Magnetron Sputtering, *L. Meng*, *H. Yu*, *T.S. Cho*, *D.N. Ruzic*, University of Illinois at Urbana Champaign

High power pulsed magnetron sputtering (HPPMS) processes, including its potential application in interconnect metallization during microfabrication, require a precise control of fluxes and energies of various plasma species. A more fundamental understanding of the pulsed discharge mechanisms and the underlying physics thus becomes necessary. In a 36 cm planar

magnetron system, a triple Langmuir probe was used to study the timeresolved behaviors of the HPPMS plasma under various discharge and pulsing parameters, as well as at different positions extending from the target surface. The pulsed plasma was shown to be established through multiple stages. And the 3-dimensional probe characterization depicted a scenario of plasma expansion from the plasma-confined racetrack, producing high density peaks up to  $10^{19}$  m<sup>-3</sup> into the pulse-off period. The delay time of these peaks from the pulse-off edge varied from several to several hundred µs with different probe positions, and was found to depend on the plasma density in the racetrack which affects the diffusion constant, and the temporal location of the peak plasma density in the racetrack as well. In addition to the probe measurements in the vicinity of the racetrack, a special setup with quartz crystal microbalance and current collecting plate behind orifices drilled in the target was designed. Ion fluxes to the cathode were directly measured, while the metal ion and argon ion fluxes were distinguished time-averagely. The self-sputtering theory of HPPMS is supported by a higher metal ion fraction measured at a higher pulse peak current. Based on the above experiments, a 1-dimensional time-dependent HPPMS discharge model was developed incorporating the surface reactions, ionization process in the plasma, and plasma diffusion. It was able to describe the essential processes of plasma buildup and dispersion, and to predict some important plasma properties such as density and ionization fraction from the basic inputs of magnetic field, discharge and pulsing parameters, etc.

# 9:20am SE+PS-TuM5 Metallic Film Modification through the Use of Non-standard HiPIMS Waveforms, *P.M. Barker, E. Lewin, J. Patscheider*, EMPA, Switzerland

High power impulse magnetron sputtering, HiPIMS, has been the source of significant scientific interest in recent years. HiPIMS consists of a high voltage / high current pulse, followed by a long off-period resulting in low duty cycles. In the present study additional control parameters are explored by using non-standard HiPIMS waveforms, consisting of pulse sequences. These pulse sequences consist of a number of micro-pulses, which can be varied in length. In a further novel approach to HiPIMS, the pulses were also applied in a bi-polar manner, with a positive voltage supplied to the same target between negative voltage pulses. Films have been grown using varied pulse sequences, in both uni- and bi-polar mode, and compared to materials grown both by traditional, single pulse, HiPIMS and dcMS.The deposition of Ti metal was chosen as a model system, and the attained coatings were analysed using X-ray diffraction, XRD, scanning electron microscopy, SEM, and X-ray photoelectron spectroscopy, XPS. An increased deposition rate, relative to a comparable standard HiPIMS pulse, was observed. The coating microstructures show increased smoothening of the coating surface and shallower surface oxidation for samples deposited using micro-pulsed HiPIMS.

10:40am SE+PS-TuM9 Reactive Modulated Pulse Power Magnetron Sputtering of Molybdenum Oxides: Optical Behavior as a Function of Process Parameters, N.R. Murphy, Air Force Research Laboratory, L. Sun, General Dynamics Information Technology, J.T. Grant, University of Dayton Research Institute, J.G. Jones, R. Jakubiak, Air Force Research Laboratory

Molybdenum oxide, a material prized for its ability to facilitate thermochromism and electrochromism, has displayed considerable sensitivity to oxygen concentrations present during reactive DC magnetron sputtering. Typical depositions performed using continuous reactive DC magnetron sputtering have demonstrated low deposition rates and limited control over stoichiometry as a result of oxygen induced target poisoning. In the following study, molybdenum oxide films were deposited using reactive modulated pulse power magnetron sputtering (MPPMS) using a 99.999% pure molybdenum sputtering target within an argon-oxygen atmosphere. At a pressure of 10 mTorr, the oxygen concentration was varied between 1 and 20%. The resulting films were characterized by way of in-situ spectroscopic ellipsometry, x-ray photoelectron spectroscopy, xray diffraction and UV-visible spectroscopy. The as-deposited films were determined to be amorphous by way of x-ray diffraction analysis and were analyzed without any prior heat treatment. In-situ spectroscopic ellipsometry was conducted throughout the deposition process, monitoring real time changes in the refractive index, extinction coefficient and thickness of the film.

11:00am SE+PS-TuM10 Cathodic Arc Plasma of AlCr Composite Cathodes in Inert and Reactive Atmospheres, *R. Franz, J. Wallig,* Lawrence Berkeley National Laboratory, *P. Polcik,* PLANSEE Composite Materials GmbH, Germany, *A. Anders,* Lawrence Berkeley National Laboratory

In the past, the distributions of ion charge states of vacuum arcs have been studied in great detail under various conditions, including their dependence on arc current level, strength of a magnetic field (if present), pressure and kind of background gases, and the distance from the plasma-producing cathode spots. Most of the work was done using pure elementary cathodes since the presence of two or more elements in the cathodes would most likely further complicate the situation when studying the physics of cathodic vacuum arcs. However, in many practical applications ternary or quaternary thin films are used. For their synthesis it is common to employ composite cathodes consisting of the elements of interest.

In the field of hard and wear-resistant coatings, thin films based on the system aluminium and chromium represent the state of the art. With the addition of nitrogen and/or oxygen ceramic coatings covering a wide compositional range can be synthesised. In the present study, a time-of-flight spectrometer was used to investigate the arc plasma composition in terms of elemental and ion charge state composition when using Al<sub>x</sub>Cr<sub>1-x</sub> ( $0 \le x \le 1$ ) composite cathodes in different gaseous environments, i.e. in vacuum as well as argon, nitrogen and oxygen atmosphere. The observed ion distributions were interpreted in the established framework of plasma generation at cathode spots and ion-gas interactions.

## Authors Index

### Bold page numbers indicate the presenter

A —
Anders, A.: SE+PS-TuM1, 1; SE+PS-TuM10, 2
B —
Barker, P.M.: SE+PS-TuM5, 1
C —
C —
Cho, T.S.: SE+PS-TuM3, 1; SE+PS-TuM4, 1
F —
Franz, R.: SE+PS-TuM10, 2
G —
Grant, J.T.: SE+PS-TuM9, 1

--- J ----Jakubiak, R.: SE+PS-TuM9, 1 Jones, J.G.: SE+PS-TuM9, 1

— **L** — Lewin, E.: SE+PS-TuM5, 1 — **M** —

Meng, L.: SE+PS-TuM3, 1; SE+PS-TuM4, 1 Murphy, N.R.: SE+PS-TuM9, 1 — P —

Patscheider, J.: SE+PS-TuM5, **1** Polcik, P.: SE+PS-TuM10, 2 **R** — R — Raman, P.: SE+PS-TuM3, 1
Ruzic, D.N.: SE+PS-TuM3, 1; SE+PS-TuM4, 1 **S** — Sun, L.: SE+PS-TuM9, 1 **W** — W — Wallig, J.: SE+PS-TuM10, 2 **Y** — Y — Yu, H.: SE+PS-TuM3, 1; SE+PS-TuM4, 1