New Horizons in Coatings and Thin Films
Room Royal Palm 1-3 - Session F2-2

HiPIMS, Pulsed Plasmas and Energetic Deposition

Moderators: Tiberiu Minea, Université Paris-Sud, Tomas Kubart, Uppsala University, Angstrom Laboratory, Sweden

1:50pm **F2-2-2 HPPMS Deposition from Composite Targets: Effect of Two Order of Magnitude Target Power Density Changes on the Composition of Sputtered Cr-Al-C Thin Films**, ***Holger Rueß,*** RWTH Aachen University, Germany*; M to Baben,* GTT-Technologies, Germany*; L Shang,* RWTH Aachen University, Germany*; P Polcik, S Kolozsvári,* Plansee Composite Materials GmbH, Germany*; M Hans,* RWTH Aachen University, Germany*; D Primetzhofer,* Uppsala University, Sweden*; J Schneider,* RWTH Aachen University, Germany

Magnetron sputtering techniques are widely used to synthesize a large number of coatings either from elemental, compound or composite targets. Coatings synthesized by direct current magnetron sputtering (DCMS) from the latter two targets often exhibit a considerable compositional deviation from the target composition, in particular, for targets containing constituents with significant mass differences [1,2]. However, for high power pulse magnetron sputtering (HPPMS), where in contrast to DCMS, a large fraction of film forming species is ionized, the magnitude of compositional deviations has not been investigated.

Hence, the effect of target power density on the composition of sputtered thin films from a Cr–Al–Cr_3C_2 composite target, as Cr, Al and C exhibit significant mass differences, was studied by utilizing DCMS (2.3 W/cm²) and HPPMS (162 and 373 W/cm²) at various substrate bias potentials and temperatures. Independent of the applied power density at the target, all Cr–Al–C thin films deposited at floating substrate potential showed no differences in composition. As the target power density was increased and a substrate bias potential was applied, aluminum deficiencies of up to 18.3 at.% were measured. Based on the measured ion currents at the substrate, preferential re-sputtering of aluminum was suggested to cause the dramatic Al depletion. As the substrate temperature was increased with an applied substrate bias potential of -100 V, the Al concentration was reduced by 8.4 at.% compared to the room temperature deposition. This may be rationalized by thermally induced desorption in addition to the afore mentioned re-sputtering effect.

[1] J. Neidhard, S. Mráz, J. M. Schneider, E. Strub, W. Bohne, B. Liedke, W. Möller, C. Mitterer, J. Appl. Phys. 104 (2008) 063304.

[2] S. Mráz, J. Emmerlich, F. Weyand, J. M. Schneider, J. Phys. D: Appl. Phys. 46 (2013) 135501.

2:10pm **F2-2-3 Adherent and Hard DLC Coatings Deposited by HiPIMS in Deep Oscillations Magnetron Sputtering (DOMS) Mode**, ***Fábio Ferreira,*** University of Coimbra, Portugal*; A Aijaz, T Kubart,* Uppsala University, Angstrom Laboratory, Sweden*; A Cavaleiro, J Oliveira,* University of Coimbra, Portugal

Diamond-like carbon coatings (DLC) make up the largest proportion of thin film coating solutions applied in the automotive industry, notably for engine applications, drivetrain components and transmission parts. DLC combine high hardness with low friction coefficient, chemical inertness, high thermal conductivity and high refractive index, making them very attractive for a wide range of other applications. In order to comply with the current trends of rising operating temperatures and lower viscosity oils, component manufacturer are currently seeking to develop a new generation of DLCs coatings, with improved properties. The main limitations of the existing DLC solutions stem from high internal stresses and limited thermal stability. The stresses, often in excess of 10 GPa, limit the maximum achievable thickness of hard H-free DLCs due to adhesion problems. H containing DLC coatings, on the other hand, have lower hardness and are typically restricted to operating temperatures below 350ºC. The main objective of this work is to develop a new generation of significantly improved well-adherent and hard DLC coatings, with high sp3/sp2 ratio and good temperature stability. DLC films were deposited by deep oscillation magnetron sputtering (DOMS), a variant of high power impulse magnetron sputtering (HIPIMS). The bombardment energy was controlled by changing the substrate bias. In order to improve the film's adhesion an interlayer was deposited. In this work, the microstructure of the films was characterized using scanning electron microscopy, Raman spectroscopy and X-ray reflectivity. The mechanical properties (Hardness and Young´s modulus) of the films were also characterized.

2:30pm **F2-2-4 Variation of Local Chemical Compositions of (Ti, Al)N Films on Inner Wall of Small Hole deposited by High Power Impulse Magnetron Sputtering**, ***Hidetoshi Komiya,*** *T Shimizu,* Tokyo Metropolitan Univeristy, Japan*; Y Teranishi, K Morikawa, M Yang,* Tokyo Metropolitan Industrial Technology Research Institute, Japan

To enhance the tool life as in industrial scale, thin films with anti-adhesive and high wear toughness are required to deposit uniformly on the three-dimensional complicated shape structure. Focusing on the great possibility of high power impulse magnetron sputtering (HiPIMS), the authors have demonstrated its availability and advantages for the industrial applications [1-3]. For the further improvement of film quality the present study focused on controlling chemical composition of thin film at inner wall of sub-millimeter scale small hole, which is specifically important for the crystal phase and mechanical properties. The small hole structure was realized by clamping the comb-shaped stainless steel plate with two flat silicon wafer substrates. To characterize the films properties at different position of inner wall depth, the several analytical techniques were performed. Surface morphology and cross-sectional microstructure of the films on the inner wall were observed using field emission scanning electron microscopy (FE-SEM). Cross-sectional SEM observation was prepared by focused ion beam (FIB). Additionally, the local chemical composition of the (Ti,Al)N films at each position of inner wall depth was analyzed by energy dispersive X-ray spectroscopy coupled with FE-SEM (SEM-EDX), which can achieve the local elemental analysis at micrometer-scaled area on the inner wall. As results, increasing tendency of atomic composition ratio of Ti and that of N / (Ti+ Al) were shown at deeper position of the inner wall. The role of ionization degree of sputtered species and its transportation and distribution into the small-hole structure are discussed by comparing with the film deposited by conventional dc magnetron sputtering.

References

[1] T.Shimizu, H.Komiya et al., *Surface and Coatings Technology*, 250 (2015) pp.44-51

[2] T.Shimizu et al., *Thin Solid Films*, 581 (2015) pp.39-47

[3] T.Shimizu, H.Komiya et al., *Thin Solid Films*, In Press (http://dx.doi.org/10.1016/j.tsf.2016.09.041)

2:50pm **F2-2-5 A Feasibility Study on the Large-area Graphene Growth by using High Power Impulse Magnetron Sputtering (HIPIMS)**, *C Pandey, M Po, D Liou, M Chen, Y Chen,* ***Ju-Liang He,*** Feng Chia University, Taiwan

High Power Impulse Magnetron Sputtering (HIPIMS), known to produce high density plasmas, is utilized to energize carbon ions from graphite substrate as cathode. for growth of large area graphene.

In this work, we aimed at depositing graphene on Copper and Silicon wafer using HIPIMS at temperatures up to 500^{o}C and substrate bias up to -800V. Vacuum pressures of 0.1 – 0.4 mTorr and deposition times of 5 minutes and 10 seconds were used. At 500^{o}C and the substrate bias set at -800V, a 7-8 layered graphene film on Copper was obtained for a deposition time of 5 seconds. An interesting feature observed using Optical Emission Spectroscopy showed that increasing the peak power increased the presence of Carbon-ion species in the plasma.

Raman spectroscopy and Raman mapping were carried out to analyze the G, D and 2D band information and I_{2D}/I_G ratios for the samples for qualitative analysis of the graphene layers respectively.

The construction of the synthesized graphene was analyzed through TEM micrographs. At zero substrate bias, amorphous carbon film was deposited on the copper substrate whereas when biased at -800V, multiple layers of graphene were deposited with a thickness of 0.33 nm .

This study showed that modifying the substrate temperature and the biasing voltage has a significant effect on the deposition of carbon atoms on the surface of copper or silicon even at low temperature of 500^{o}C as compared to the high temperatures used in CVD techniques.

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