Wednesday Afternoon, April 26, 2017

Hard Coatings and Vapor Deposition Technologies
Room Golden West - Session B3-1

Deposition Technologies and Applications for Diamond-like Coatings

Moderators: Frank Papa, Gencoa Ltd., USA, Klaus Böbel, Bosch GmbH

2:10pm **B3-1-3 New Pathways for Improving Adhesion of DLC on Steel in Low Temperatures***, L Leidens,* UCS and CAPES, Brazil*; Â Crespi,* UCS, Brazil*; F Alvarez,* IFGW-UNICAMP, Brazil*; **Carlos Figueroa**,* UCS, Brazil

Diamond-like carbon thin films (DLC) are state-of-art coatings that can have properties which are object of interest such as ultra-low friction coefficient, chemical inertness and low wear rates. Despite of its strident properties, the use of DLC is not yet fully widespread due to the poor adhesion of the film in some substrates, for example, plain and low-alloy steels. In order to improve DLC adhesion on steel, different interlayers have been proposed. On one hand, hybrid technologies containing a step of PVD deposition of a metal/metal nitride interlayer are industrially used, although its costs are high for several applications. On the other hand, PECVD technologies can produce silicon-containing interlayers, although the deposition temperatures that prompt adhesion as high as 300ºC. Previous works have pointed out that oxygen atoms act as terminator species in silicon-containing interlayers that diminish DLC adhesion on steel.

The aim of this work is to investigate new pathways of reaching high DLC adhesion by using silicon-containing interlayers in low deposition temperatures. In order to explore alternatives, a hydrogen plasma etching effect was analyzed in two different set of samples. [3] The first set look at analyzing the physicochemical processes due to hydrogen plasma interaction in the silicon-containing interlayer and, consequently, the interlayers were deposited with HMDSO at a constant temperature and time of 300°C and 10 min, respectively, varying the hydrogen etching time (0 to 10 min) at 85°C on AISI 4140 low-alloy steel. The second set look at analyzing the adhesion in low deposition temperatures and, consequently, the interlayers were obtained at different deposition temperatures (80 to 180°C) and the hydrogen plasma etching was performed at a constant temperature and time of 85°C and 6 min, respectively.

The samples were characterized by SEM, GDOES, adhesion tests, and profilometry. One can see that the interlayer thickness decreases with the increasing of the hydrogen plasma etching processing time. The etching mechanism suddenly changes at a processing time of 4 minutes. Whereas shorter etching times than 4 min remove roughly constant contents of carbon, silicon and oxygen, longer etching times than 4 min remove more silicon and oxygen than carbon. Moreover, the hydrogen etching process prompts to good adhesion of the DLC on low-alloy steel as low temperature as 85ºC. We propose a mechanism where the outermost interface (DLC/interlayer) is constituted by more carbon than silicon and oxygen atoms after the hydrogen etching process enabling more carbon-carbon chemical bonds than before, which increase the adhesion.

2:30pm **B3-1-4 Stress Evolution of Diamond-like Carbon Films via Controlled Metal Doping***, **Aiying Wang**, X Li, L Sun, P Guo,* Ningbo Institute of Materials Technology and Engineering, Chinese Academy of Sciences, China

>It is well known that, being one family member of carbon-based solid materials, diamond-like carbon (DLC) films own more comprehensive advantages over diamond, carbon nanotube, even as well as very recently the blooming graphene, due to their superior physiochemical properties, facile synthesis with low cost over large uniformity, and amorphous smooth surface etc. In particular, DLC films enable the possibility to apply the eco-friendly surface engineering to satisfy the strong demands of energy saving and emission reduction to update. In this talk, we will introduce our study on the stress controlling of DLC films via metal doping, where the feature and concentration of doped metal were controlled. Specifically, the mechanism to understand the stress reduction originated from the various doped metals will be addressed in view point of combined experimental and theoretical studies. Further synergistic effect of co-doped metals on the microstructure and properties of Me-DLC films will be discussed, which provide a new concept to fabricate the DLC films with comprehensively high performance for wide range of industrial applications.

2:50pm **B3-1-5 Influence of Alloying Metals on Tribological Properties of Diamond-like Carbon Films Synthesized by Metal Plasma Activated Deposition Process***, D Wang, **Wei-Yu Ho**, M Shih, W Chen,* MingDao University, Taiwan*; J Wang, J Hung,* Aurora Scientiftic Corp, Canada

The metal plasma activated diamond-like carbon (mpa-DLC) film has been commercially adopted by manufacturing industries for its superior tribological and functional properties to prolong tools and components service life. The multi-layered and gradient mpa-DLC coating consists of a metallic interface layer, a metal nitride/carbide transition layer and the outmost DLC film, which was synthesized by breaking down of acetylene feed gas. The whole process can be categorized as an integrated and PVD-activated CVD synthesis of DLC coatings. TiN and CrN are among the common starting and supporting materials between the substrate and the top DLC layer. This research looked into the strengthening mechanism of the interface bonding and the intrinsic tribological property of the mpa-DLC coating by adding second and/or third metal elements such as Al, Cr, or Si to the TiN and CrN base lattice. The electron states of carbon and the chemical bonding configuration will be studied by using ESCA-SIMS. The tribological properties will be investigated by using ball-on-disk tribo-meter, nano-indentation, and scratch tester. The thermal property will be examined by using TG/DT. Results of this research will provide a mechanism to further improve the mpa-DLC coatings.

Keywords: metal plasma, DLC, tribology, PVD coating

3:10pm **B3-1-6 Thick Diamond Like Carbon Coatings Deposited by Deep Oscillation Magnetron Sputtering for Automotive Applications***, **Jianliang Lin**, P Lee, R Wei, K Coulter,* Southwest Research Institute, USA

Hydrogenated diamond like carbon (DLC) coatings with thicknesses up to 20 µm have been deposited by sputtering a graphite target in Ar and acetylene (C_2H_2) mixture using a two-step process including plasma enhanced magnetron sputtering (PEMS) and deep oscillation magnetron sputtering (DOMS). The PEMS technique was used for substrate ion cleaning and sputter deposition of the Ti/TiN bond layer, while the DOMS technique was used to sputter graphite in an Ar+C_2H_2 environment to form the top DLC layer. The effects of the C_2H_2 flow rate (f_{C2H2}) on the deposition rate, adhesion, surface roughness, mechanical and tribological properties of the coatings were studied by means of scanning electron microscopy, Raman spectroscopy, HRC indentation, nanoindentation, dry ball-on-disk test, and block-on-ring test in SAE 10W-30 engine lubricant. Appropriately introducing C_2H_2 (e.g.10 sccm, 4% of the total gas flow) increased the deposition rate, the sp^3/sp^2 ratio, and hardness and wear resistance of the DLC coatings. In contrast, higher C_2H_2 flows (>30 sccm, 12% of total gas flow) showed detrimental effects on the adhesion as well as the mechanical properties of the coatings. These thick hydrogenated DLC coatings exhibited low dry sliding COF in the range of 0.07 to 0.12 in the ambient air, and low friction in SAE 10W-30 engine lubricant in the range of 0.08 to 0.090.

3:30pm **B3-1-7 Deposition of ta-C by Filtered and Unfiltered Laser-arc Technique – Actual Status***, **Volker Weihnacht**, G Englberger, A Leson,* Fraunhofer IWS, Germany **INVITED**

Due to their unique combination of superhardness and low friction properties tetrahedral amorphous carbon films (ta-C) are very attractive as tribological coatings e.g. on automotive sliding components like piston pins, piston rings, and valve-train parts. In contrast to conventional DLC films (a-C:H), ta-C cannot be deposited by PECVD or magnetron sputtering techniques. The only effective technique for mass production of ta-C coatings is arc evaporation of graphite. As the arc spot movement on graphite is very difficult to control, a laser triggered pulsed arc evaporation technique was developed at Fraunhofer IWS.

This contribution will show how the laser-arc technique has developed from an idea and first prototypes to an industrially upscaled technology used for mass production of ta-C coatings. Special emphasis will be put on the issue of deposition rate versus ta-C coating properties and possibilities how to modify the coating architecture to deposit well-adhering ta-C coatings with thickness of even more than 10 µm.

Furthermore, the issue of arc-induced particle emission and defects in the ta-C coatings will be addressed. It will be shown how plasma filtering specifically developed for the laser-arc technique will suppress the particle transmission from the cathode to the substrate and hence reduce the defect density and roughness of deposited coatings.

4:10pm **B3-1-9 Wear Behavior of CoCrMo Alloy Coated with Highly Adhesive N-Doped DLC by ICP-CVD**, ***Jesus Corona Gomez***, *Q Yang,* University of Saskatchewan, Canada

Coating the joint surfaces with diamond like carbon (DLC) is a promising way to increase the service lifetime of hip joints made of CoCrMo alloy. However, the weak adhesion of DLC on the alloy presents problems for this application. This work aims to improve the adhesion of DLC on CoCrMo alloy with Nitrogen doping and nanodiamond incorporation. Nanodiamond particles with different densities was synthesized on CoCrMo alloy sheets by microwave plasma enhanced chemical vapor deposition and nitrogen doped DLC thin films were then deposited on them by Inductively Coupled Plasma assisted Chemical Vapor Deposition (ICP-CVD). The effect of nitrogen doping and nanodiamond incorporation on the film adhesion was investigated by scratch testing and wear behavior of the samples were studied using ball-on-disc testing. Results showed that Nitrogen doping and nanodiamond incorporation could improve the film adhesion significantly and thus the wear performance. The excellent tribological performance of the coated samples is attributed to the graphitization of the top layer of the DLC according to the Raman spectrum analysis. The wear resistance of the DLC coated CoCrMo alloy was superior to the bare CoCrMo alloy due to the high adhesion of DLC on the alloy. The results have demonstrated that the modified DLC films are promising for total hip joint replacement application.

4:30pm **B3-1-10 Carbon-Based Coatings on Nanofabric by Using HIPIMS for Possible EAOPs Applications**, ***Pi-Wei Wang***, *C Tsen, C Liu, J He,* Feng Chia University, Taiwan

Nanofibers, capable of providing extra functions in contrast to regular-size fibers due to vary high specific surface area. This has brought a large opportunity for the nanofibers to utilize in form of nanofibrous membranes as air and water filtration materials. In combination with advanced coating technique such as high power impulse magnetron sputtering (HIPIMS), it would be able to develop new form of filtration materials for better purifying performance. In this study, carbon-based coatings were deposited on nanofibrous membranes by using HIPIMS (where high density plasma favorites sp3-containing carbon film growth at low-temperature) to achieve additional functions for use as membrane electrode of an electrochemical advanced oxidation processes (EAOPs). Experimental results show that mixed sp2/sp3 carbon film can be obtained, through the result of Raman spectroscopy analysis. The deposited nanofibrous membrane can be electrical conductive for EAOPs purpose. Degradation efficacy for specific organic substance can vary depending on the deposition parameters and were discussed.

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