Hard Coatings and Vapor Deposition Technologies
Room California - Session B3-1-MoM

Deposition Technologies and Applications for Diamond-like Coatings I

Moderator: Klaus Böbel, Bosch GmbH

10:20am **B3-1-MoM-2 On the Deposition and Properties of Carbon-based Multilayer Systems Prepared by PLD**, ***Steffen Weißmantel,*** University of Applied Sciences Mittweida, Germany; *M Hess,* Fritz Stepper GmbH & Co. KG, Deutschland, Germany; *R Bertram, D Haldan, T Warnk, J Maus, S Rupp,* University of Applied Sciences Mittweida, Germany

The layer deposition technique Pulsed Laser Deposition (PLD) provides a feasible way to produce pure carbon films in a wide range of mechanical properties. These properties cover, depending on the deposition parameters, indentation hardness H_{IT} from 20 GPa up to 70 GPa and indentation modulus E_{IT} from 300 GPa up to 700 GPa. As we are going to show, the variation of these mechanical properties over wide ranges can be correlated with the Raman spectra of the films. In particular, the intensity ratio of the disordered and graphitic peak provides an efficient way to determine the mechanical properties of the hydrogen free amorphous carbon films.

Based on the fact that the variation of hardness can be done simply by varying the laser fluence, layered structures consisting of sublayers of alternating or continuously changing sp^3 content and hardness were deposited. The periodicity of the bilayer stacks were varied from 500 nm down to 2.5 nm resulting in an immense increase of interfaces up to 800 since the total film thickness was kept constant at 2 micron. Keeping the ta-C film component constant at some 65 GPa , the hardness of the soft a-C layer component has been changed in the range of 20 up to 50 GPa. The toughness and resistivity against wear and cracking of these multilayered films were evaluated and compared to super-hard single ta-C films. We could show that properly designed multilayered structures have in this respect much improved properties compared to the single layer. In scratch tests, these multilayers show besides an excellent adhesion to various substrate materials a significantly improved, very high cohesive breaking strength. In addition, by testing the abrasive wear of such coating systems against polycrystalline diamond suspension using calotte grinding and against various ceramics and metals using a pin-on-disk tribotester, superior durability was identified, surpassing conventional wear protection layers by up to 3 orders of magnitude.

A computational analysis of the stress distribution in the film substrate systems was used to get an idea of the positions and values of stress under different load scenarios. Based on that film architectures were designed and deposited that show optimized stress distributions to relieve the strain at the substrate layer-system interface. It will be shown that a proper film design enables the deposition and application of extremely hard, durable and yet tough diamond like carbon coating systems. These outstanding layer properties such as high hardness, elasticity, toughness and wear resistance show the great potential of such carbon-based films, i.e. for application as wear protection coatings.

10:40am **B3-1-MoM-3 Improved Adhesion of a-C and a-C:H Films with a CrC Interlayer on 16MnCr5 by HiPIMS-Pretreatment**, *W Tillmann,* ***Nelson Filipe Lopes Dias,*** *D Stangier,* TU Dortmund University, Germany; *W Maus-Friedrichs, R Gustus,* Technical University Clausthal, Germany

A high adhesion of amorphous carbon films to steel substrates remains a challenging task, sustaining continuous research efforts to improve the adhesion strength. Besides the interlayer system and the substrate material, surface pretreatments have a crucial role on the adhesion behavior. Within this context, the influence of the High Power Impulse Magnetron Sputtering (HiPIMS) pretreatment on the adhesion of hydrogenfree (a-C) and hydrogenated (a-C:H) amorphous carbon films with a chromium carbide (CrC) interlayer on 16MnCr5 steel is investigated. The plasma treatment consisted of 30 min Ar ion etching as well as a sequential 5 min of HiPIMS-pretreatment with a Cr cathode, subsequently comparing this procedure to a procedure without the HiPIMS technique. The impact of the HiPIMS-pretreatment on the structure of the film was systematically analyzed by taking the CrC interlayer as well as the entire film structure into consideration.

The adhesion strength of the a-C and a-C:H films is significantly improved by HiPIMS-pretreating the 16MnCr5 steel. In scratch tests, the critical load L_{c3} for a total film delamination increases from 43 ± 4 to 59 ± 3 N and from 48 ± 2 to 64 ± 3 N for the a–C and a–C:H film. The improved adhesion behavior of the carbon films is ascribed to the increased adhesion of the CrC interlayer, which did not delaminate when scratched with a load up to 159 ± 18 N . Complementary Rockwell indentation tests reveal that the HiPIMS-pretreatment improves the adhesion class from HF6 to HF4 and from HF5 to HF3 for a–C and a–C:H. The enhanced adhesion is essential to exploit the properties of a–C and a–C:H films in applications with high loads. In conclusion, the HiPIMS-pretreatment has proven to be a promising technique to increase the adhesion strength of carbon films.

11:00am **B3-1-MoM-4 Properties Of Diamond-Like Carbon Films With Incorporated CVD-Diamond Nanoparticles**, ***Rebeca Falcão,*** Institute of Science and Technology, Federal University of São Paulo (UNIFESP), Brasil; *C Wachesk,* Federal University of São Paulo, Brazil, Brasil; *T Taiariol,* National Institute for Space Research, Brazil; *G Vasconcelos,* Instituto de Estudos Avançados, Brazil; *E Corat, V Trava-Airoldi,* National Institute for Space Research, Brazil

Diamond-like carbon (DLC) films have been extensively applied as a surface coating due their attractive mechanical, chemical and tribological properties. The properties of the a-C:H films can be significantly enhanced by the presence of diamond nanoparticles in their structure with some apparent advantages by combining hardness, low roughness, coefficient of friction, biocompatibility, etc., of both materials. However, functionalization of diamond nanoparticles and their severe big clusters formations represent some challenges to be overcome. Therefore, the innovative aspect of this work is the growth of DLC films with incorporated CVD nanodiamonds (NDs), obtained by high energy ball milling technique with controlled sizes and functionalization avoiding a lot of clusters formation. In this work, CVD NDs, obtained at the first time, were chemically processed due to the high levels of contamination by using fluoric and nitric acid chemical attack. The DLC films were deposited on a metallic substrate by using a modified Pulsed DC Plasma-Enhanced Chemical Vapor Deposition (PECVD) technique, and the incorporation of the NDs into the DLC films structure was carried using a colloidal solution of NDs and DLC films precursor. The influence of the CVD NDs size on physical and chemical properties of the hybrid film, such as hardness, coefficient of friction, morphology, and chemical inertia were investigated. Nanoparticles size and the level of purity was analyzed by dynamic light scattering and X-Ray diffractometry (XRD) technique, respectively. The hybrid DLC films were characterized by scanning electronic microscopy-field emission gun, XRD and Raman scattering spectroscopy. Also, the qualitative adhesion of the film was analyzed by RHC 1500 N indentations in accordance with the VDI3198 standard. An apparent application with preliminary results is also a part of this work.

Acknowledgments:

The authors want to thanks to the Fundação de Amparo à Pesquisa do Estado de São Paulo – FAPESP (grants n. 2017/08899-3 and 2012/15857-1), CNPq and Capes.

11:20am **B3-1-MoM-5 Influence of the Argon as an Ignitor and an Agent on DLC Properties Growth at Pressure as Low as 3 x 10^{-4} mbar by Modified Pulsed-DC PECVD Method**, ***Vladimir Jesus Trava-Airoldi,*** *K Nass, E Corat,* National Institute for Space Research, Brazil; *N Fukumasu,* Sao Paulo University, Brazil; *M Ramirez,* University of Vale do Paraiba, Brazil; *G Capote,* National University of Bogota, Colombia

As reported for many years, hydrogenated DLC films (a-C:H) have been a choice of a protective coatings for many applications due to their set of superior mechanical, chemical, tribological and biological properties such as: high hardness, high wear resistance, low coefficient of friction, high chemical inertness, good biocompatibility, bactericide, etc.. However, in order to improve the adhesion between the DLC and the different metals substrates a huge modification of a Pulsed DC PECVD technique has been obtained introducing an additional cathode working as electron and ion confinement. With these modifications pressure as low as 10^{-3} mbar allow operating in collisionless regime improving not only the adhesion but also a set of DLC proprieties cited above. So, in this work we present studies concerning more improvement properties of the DLC films as a function of confinement of electrons and ions parameters in a plasma discharge by using argon as an ignitor gas for the interlayer precursor and keeping it during all the process of DLC deposition. In this case the best conditions of collisonless operation was reached at pressure as low as 3.10^{-4} mbar. Basically, due to the condition of operating in very low pressure, this technique allows to grow the DLC film with very good uniformity and higher hardness, higher adhesion, lower coefficient of friction, less porosity and, also, provide to be able to get a DLC deposition in the form of multilayer, like thicker films, promoting less residual stress. So, studies at the first time, of DLC with superior properties has been carried out from PECVD technique. More specifically, studies of the DLC film properties as a function of the argon buffer gas density and as a function of bias voltage has been done. Raman scattering spectroscopy, Rockwell indentation, nano indentation, FEG, and trybological analyses are discussed. Also, the operating parameters of this modified PECVD system are well controlled, so that a scaling up studies will also be presented as an important part of this work.

Keywords: DLC films; DC pulsed PECVD; additional cathode; argon ignitor, mechanical and trybological properties.

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