Hard Coatings and Vapor Deposition Technologies
Room On Demand - Session B3

Deposition Technologies and Applications for Diamond-like Coatings

B3-1 'Hydrogenated Amorphous Carbon from Magnetized Hollow Cathode Discharges, ***John Miller (miller290@llnl.gov)****, A. Ceballos-Sanchexz, S. Elhadj, S. Kucheyev, B. Bayu Aji, S. Falabella,* Lawrence Livermore National Laboratory, USA

Diamond-like carbon is a popular hard coating material whose properties widely depend on deposition process and conditions within the process. Variations between the methods and conditions yield films with varying hydrogen, sp2 carbon, and sp3 carbon content who have densities between 1 and 3 g/cc, spanning the difference between a low-density hydrocarbon polymer and polycrystalline diamond (high density carbon). This material is becoming increasingly interesting as a new ablator material for inertial confinement fusion because it is low Z and has the potential to tune its density between that of glow discharge polymer(~1 g/cc) and diamond (3.5 g/cc), two ICF ablator materials of choice. This work investigates the use of a magnetized hollow cathode discharge for depositing thick, density tunable amorphous carbon films for future inertial confinement fusion experiments.A variety of the properties of the films will be discussed including: composition, density, impurity content, thickness limitation, deposition rate and surface morphology.

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B3-2 Effect of Mechanical and Thermochemical Tool Steel Substrate Pre-treatment on DLC Coating Durability, ***Daniel Tobola (daniel.tobola@kit.lukasiewicz.gov.pl)****,* Łukasiewicz Research Network - Krakow Institute of Technology, Poland*; T. Liskiewicz,* Manchester Metropolitan University, UK*; L. Yang,* Leeds University, UK*; T. Khan,* Manchester Metropolitan University, UK*; Ł. Boron,* Łukasiewicz Research Network - Krakow Institute of Technology, Poland

DLC coatings are becoming well established across many industrial sectors including aerospace, automotive, oil and gas, and cold forming tools. While DLC coatings exhibit good mechanical properties and low coefficient of friction for themselves, DLC coating–substrate systems may suffer from insufficient wear resistance. This paper describes the wear mechanisms and reports characteristics of the DLC coating-steel substrate system behaviour after mechanical and thermochemical steel substrate pre-treatments. We have investigated two tool steel substrates, Sverker 21 (AISI D2) and advanced powder metallurgy alloyed steel Vanadis 8. Initially, the substrates were heat treated in vacuum furnace and gas quenched resulting in hardness of 59±1 and 64±1 HRC respectively. Subsequently the samples were subjected to mechanical turning and burnishing with 130N and 160N forces, using diamond composite tools with ceramic bonding phase. Afterwards, a vacuum nitriding process in a PVD chamber as a pre-treatment for subsequent DLC coating deposition was carried out. Coated samples were subjected to a series of ball-on-disc wear tests against Al2O3 and Si3N4 counterparts. X-ray diffraction (XRD), instrumented indentation and scanning electron microscopy (SEM) were employed to examine the phase composition, nano-hardness, Young's modulus, surface microstructure, elemental distribution and 3D surface topographies of the wear scars. Selected variable factors including the type of steel, the burnishing force, type of counter-body material were subjected to statistical analysis. The effect of sequential processes used as pre-treatment on DLC coating durability was demonstrated and the results are discussed in light of improving the cold forming tools tribological performance.

Keywords: DLC coatings, tool steels, vacuum nitriding, slide burnishing

B3-3 Stress Free ta-C Coatings by means of Up-Scaled Pulsed Laser Deposition for Industrial Applications, ***Hagen Gruettner (hagen.gruettner@hs-mittweida.de)****, D. Haldan, J. Maus,* Hochschule Mittweida - University of Applied Sciences, Germany*; M. Nieher,* Hochschule Mittweida - University of Applied Sciences , Germany*; S. Weissmantel,* Hochschule Mittweida - University of Applied Sciences, Germany

In many industrial applications highly wear-resistant coatings are required to protect and / or functionalize tools and components. While typical representatives of DLC coatings are already in large-scale industrial use, stress free ta-C coatings produced at the Laser Institute Mittweida (LHM) by means of a patented combination of pulsed laser deposition and pulsed laser stress relaxation are currently well under way due to their outstanding properties and the possibility to up-scale the deposition process. These ta-C coatings are special representatives of the DLC coatings and unsurpassed in terms of their hardness and usability. Moreover, they can be prepared almost free of intrinsic stress. Using optimal deposition parameters, a hardness of up to 70 GPa with Young's moduli of 700 to 800 GPa can be achieved, which leads to an extremely high operational wear resistance. Due to the low average surface roughness (Ra) of a few nanometers and the low friction coefficient these layers are also excellently suitable for tribological applications. By using optimized adhesion layers or layer systems it is possible to achieve high adhesion strengths on a variety of substrate materials. The low temperature required during the deposition process (< 90 °C) makes it also possible to coat temperature-sensitive materials. Due to their chemical resistance, biocompatibility and dopability the ta-C coatings offer a wide range of further applications, such as in medical technology, food industry or sensor technology. By means of the combined pulsed laser deposition and laser pulse annealing process the LHM is able to produce homogeneous layers with a thickness ranging from a few nanometers up to a few 10 µm with an adjustable hardness between 25 and 70 GPa and an adjustable intrinsic stress between 12 GPa and nearly zero GPa by choosing the corresponding deposition parameters. That makes it also possible to prepare material and application specific layer designs like Young's modulus gradients or multilayers. It will be shown that the deposition process can be up-scaled to industrial requirements using new high power excimer lasers, which were introduced in 2019. The LHM is now looking for interested partners and potential customers.

B3-4 DLC Coatings Deposited by Novel Doping Strategies with HiPIMS, ***José Antonio Santiago Varela (joseantonio.santiago@nano4energy.eu)****, I. Fernández-Martínez, A. Wennberg,* Nano4Energy SL, Spain*; M. Monclús, J. Molina-Aldareguia,* IMDEA Materials Institute, Spain*; V. Bellido-Gonzalez,* Gencoa Ltd, UK*; M. Panizo-Laiz,* Universidad Politecnica de Madrid, Spain*; J. Sánchez-Lopez, T. Rojas,* CSIC, Spain*; S. Goel,* Cranfield University, UK*; J. Endrino,* IKERBASQUE, Spain

Diamond-like Carbon (DLC) coatings have been recognized as one of the most valuable engineering materials for various industrial applications including manufacturing, transportation, biomedical and microelectronics. Among its many properties, DLC stands out for a good frictional behaviour combined with high surface hardness, offering an elevated protection against abrasive wear. Nevertheless, a factor limiting the widespread application of DLC coatings is their thermal stability. DLC is very temperature-sensitive since its sp^3-sp^2 structure undergoes a graphitization process at high temperatures that deteriorates both hardness and coefficient of friction. In order to overcome this limitation, new ways to modify DLC coatings for acceptable high temperature performance have been explored. In this work, we investigated a novel deposition technique of hard DLC coatings doped with various elements (e.g. W, Cr, Ti, Si) using HiPIMS by incorporation of positive pulses. Highly ionized plasma discharges were obtained during HiPIMS deposition. The high ion energy bombardment resulted in a higher sp^3 to sp^2 bond ratio. EELS and Raman spectroscopy were used to characterize the sp^3 and sp^2 structures in the deposited films. Nanoindentation tests revealed improved mechanical properties (hardness up to 35 GPa) for doped DLC coatings. Additional high temperature nanoindentation tests performed in the range of 27 ºC to 450 ºC showed that the mechanical properties at high temperature are dependent on the sp^3 content. Pin-on-disk tests were carried out in order to assess the tribological performance of the coatings both at room and high temperature. The increased toughness and reduced compressive stress that doping provides to the carbon matrix together with a high sp^3 bonding structure obtained with HiPIMS deposition improves the stability of DLC coatings for high temperature tribological applications. Finally, micromilling trials were carried out to assess the performance of these doped DLC coatings in micromachining of Ti6Al4V samples and compared to an uncoated tool, an increased tool performance was obtained.

B3-5 Preparation of Hybrid ta-C/MoS_2-Films by using Laser Arc Technology, ***Frank Kaulfuss (frank.kaulfuss@iws.fraunhofer.de)****, F. Hofmann, Y. Han, F. Schaller,* Fraunhofer IWS, Germany*; T. Kruelle,* Fraunhofer IWS,Germany*; S. Makowski, V. Weihnacht, A. Leson, L. Lorenz, M. Zawischa,* Fraunhofer IWS, Germany

Hydrogen-free ta-C coatings are already used to reduce friction in lubricated environments. The application under minimum quantity lubricated and non-lubricated boundary conditions remains a great challenge. The addition of the dry lubricant MoS_2 is intended to improve the performance characteristics of the ta-C in this case. For the production of ta-C coatings, the Laser Arc process is particularly suitable, with which very hard and low-defect coatings can be produced. MoS_2 targets can be combined and alternately evaporated using the same technique.

By adapting the discharge conditions, ta-C/MoS_2 layers could be produced in different variants. In addition to ta-C with simple MoS_2 top layer, multilayers with alternating deposition were also produced with single layer thicknesses in the nanometer range. The plasma states of the components, which have a large influence on the layer formation, were investigated as well as the cathode spot behaviour at the different materials (graphite, MoS_2). The investigations also concentrated on the mechanical properties of the layers, which were determined by SEM, TEM, X-ray diffraction, nanoindentation and scratch testing. In addition, tribological tests provide information on the influence of the layer structure in different applications.

B3-6 Effects of Target Poisoning Ratios on the Microstructure, Mechanical Properties and Corrosion Resistance of WC_x Coatings Fabricated by Superimposed HiPIMS and MF System*, **Igamcha Moirangthem**,* Ming Chi University of Technology, Taiwan*; S. Chen,* National Taiwan University, Taiwan*; J. Lee (jefflee@mail.mcut.edu.tw),* Ming Chi University of Technology, Taiwan

In this work, we studied the effect of target poisoning ratios of tungsten carbide films using a superimposed HiPIMS and MF system. The flow rate of the acetylene (C_2H_2) was controlled using a plasma emission monitoring (PEM) system to feedback control the target poisoning ratio during deposition. Five coatings, WC10, WC30, WC50, WC70 and WC90, were grown under target poisoning ratios of 10%, 30%, 50%, 70% and 90% respectively, on Si, AISI420 stainless steel (SS) and AISI304 SS substrates. A small hysteresis loop area for the emission intensity of W at 429.6 nm was observed with variation of C_2H_2 gas flow rates. With increasing target poisoning ratios, the phases changed from nanocrystalline β-WC_{1-x} to amorphous. The X-ray photoelectron spectroscopy was used to study the chemical bindings of coating. The highest power normalized deposition rate of 25.20 nm $min^{-1}kW^{-1}$ was observed for WC90. The highest hardness value of 32.3 GPa was measured for WC30. WC50 showed the best adhesion among the coatings with L_{C2} value of 65.1 N. The lowest COF of 0.26 was observed for WC90. In potentiodynamic polarization test using 0.1M H_2SO_4 solution, WC90 showed colourful fringes around the corrosion micro-pits with the highest polarization resistance (R_p) of 4552.51 kΩcm^2.

Keywords: Superimposed high power impulse magnetron sputtering, middle frequency, WCx coatings, tungsten doped diamond-like carbon coating, target poisoning, nanoindentation, tribometer

B3-7 The Influence of Different Power Supply Systems on the Microstructure, Mechanical and Corrosion Properties of Titanium Carbide Coatings*, H. Yu -Tung,* Ming Chi University of Technology, Taiwan, Republic of China*; C. Li-Chun,* Ming Chi University of Technology, Taiwan*; L. Bih-Show,* Chang Gung University, Taiwan*; **Lee Jyh-Wei (jefflee@mail.mcut.edu.tw)**,* Ming Chi University of Technology, Taiwan

Titanium carbide (TiC) coatings have attracted wide attention from researchers and industry due to their high hardness, good wear and corrosion resistance. In this study, the TiC coatings were fabricated by four different power supply systems including superimposed HiPIMS-MF, HiPIMS, MF, and DC in a gas mixture of acetylene and argon. During the deposition process, a plasma emission monitoring (PEM) system was used to control the Ti target poisoning status to 70%. The single crystal silicon wafers and AISI304 stainless steel plates were used as substrates. The chemical compositions of TiC films were analyzed by a field emission electron probe microanalyzer. The microstructure of thin film was examined by a field emission scanning electron microscope. The crystalline structure of thin film was analyzed by an X-ray diffractometry. The hardness, adhesion and tribological properties of TiC films were evaluated by nanoindenter, scratch test and pin-on-disk wear test, respectively. The corrosion resistance of TiC films in 0.1 M H_2SO_4 aqueous solution was examined by an electrochemical workstation. The influence of four different power supplies on the deposition rate, microstructure, hardness, adhesion, wear and corrosion resistance of TiC films were studied in this work. Although the deposition rate of the TiC coating deposited by DC power supply was the highest, the film quality was inferior to other films due to its less dense microstructure. On the other hand, the TiC coating fabricated by superimposed HiPIMS-MF power supply exhibited a dense microstructure, good mechanical property and excellent corrosion resistance.

Author Index

Bold page numbers indicate presenter

— B —
Bayu Aji, B.: B3-1, 1
Bellido-Gonzalez, V.: B3-4, 1
Bih-Show, L.: B3-7, 2
Boron, Ł.: B3-2, 1
— C —
Ceballos-Sanchexz, A.: B3-1, 1
Chen, S.: B3-6, 2
— E —
Elhadj, S.: B3-1, 1
Endrino, J.: B3-4, 1
— F —
Falabella, S.: B3-1, 1
Fernández-Martínez, I.: B3-4, 1
— G —
Goel, S.: B3-4, 1
Gruettner, H.: B3-3, **1**
— H —
Haldan, D.: B3-3, 1
Han, Y.: B3-5, 1
Hofmann, F.: B3-5, 1
— J —
Jyh-Wei, L.: B3-7, **2**
— K —
Kaulfuss, F.: B3-5, **1**
Khan, T.: B3-2, 1
Kruelle, T.: B3-5, 1
Kucheyev, S.: B3-1, 1
— L —
Lee, J.: B3-6, 2
Leson, A.: B3-5, 1
Li-Chun, C.: B3-7, 2
Liskiewicz, T.: B3-2, 1
Lorenz, L.: B3-5, 1
— M —
Makowski, S.: B3-5, 1
Maus, J.: B3-3, 1
Miller, J.: B3-1, **1**
Moirangthem, I.: B3-6, **2**
Molina-Aldareguia, J.: B3-4, 1
Monclús, M.: B3-4, 1
— N —
Nieher, M.: B3-3, 1
— P —
Panizo-Laiz, M.: B3-4, 1
— R —
Rojas, T.: B3-4, 1
— S —
Sánchez-Lopez, J.: B3-4, 1
Santiago Varela, J.: B3-4, **1**
Schaller, F.: B3-5, 1
— T —
Tobola, D.: B3-2, **1**
— W —
Weihnacht, V.: B3-5, 1
Weissmantel, S.: B3-3, 1
Wennberg, A.: B3-4, 1
— Y —
Yang, L.: B3-2, 1
Yu -Tung, H.: B3-7, 2
— Z —
Zawischa, M.: B3-5, 1