Wednesday Afternoon, April 25, 2018

Hard Coatings and Vapor Deposition Technologies Room California - Session B3

Deposition Technologies and Applications for Diamond-like Coatings

Moderators: Frank Papa, Gencoa, Konrad Fadenberger, Robert Bosch GmbH

1:30pm B3-1 Tribology of Diamondlike Carbons in Various Application Environments, Gary Doll, University of Akron, USA INVITED Hydrogen free and hydrogen containing amorphous carbon materials are commonly referred to as diamondlike carbons. Due to the hardness and the ability to deposit coatings at temperatures compatible with most engineering materials, diamondlike carbons have become widely used as tribological coatings for addressing friction and wear in mechanical components. There are few if any intrinsic properties of these synthetic materials since their mechanical, structural, and compositional properties are strongly dependent upon the deposition process conditions. Furthermore, a high level of fundamental knowledge on how material properties relate to the tribological performances of diamondlike carbon coatings in various application environments has not been achieved. Application environments can be categorized by the type of tribological contact (rolling, sliding, mixed mode), lubrication condition, temperature, atmosphere, loading, and other items. This presentation shall discuss several examples where subtle differences in the deposition processes of diamondlike carbon coatings were responsible for transitioning unacceptable tribological performances in specific application environments to acceptable ones.

2:10pm **B3-3 Synthesis and Comparison of Highly Tetrahedral Amorphous Carbon by Arc-mixed HiPIMS and Arc-free HiPIMS Modes**, *H Hug, Rajesh Ganesan*, *K Thorwarth*, EMPA Swiss Federal Laboratories for Materials Science and Technology, Switzerland; *M Tucker*, *N Marks*, Curtin University, Australia; *M Stüber*, *S Ulrich*, Karlsruhe Institute of Technology (KIT), Germany; *D McKenzie*, *M Bilek*, The University of Sydney, Australia; *S Guimond*, *M Arndt*, Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein

Tetrahedral amorphous carbon films with high sp³ content have been synthesized by high power impulse magnetron sputtering (HiPIMS) operated in an arc-free sputtering mode and in a mixed sputtering/arc mode. The properties of the films deposited in these both modes have been compared. In the mixed-mode, the presence of macroparticles in the deposited films were observed, though the amount and size of macroparticles are less than that of the ones in the films deposited by filtered cathodic arc vapour deposition. The short-lived cathode spots form in the magnetic racetrack and produce large numbers of carbon ions in the mixed-mode HiPIMS. In the arc-free HiPIMS, the macroparticle content in the films have been significantly reduced, however the deposition process has to be tuned for high degree of carbon ion production in order to compensate the loss of arc-facilitated carbon ion generation.

2:30pm **B3-4 Evaluation of Superhard ta-C Coatings for the Machining of Synthetic Materials,** *Frank Kaulfuss,* Fraunhofer Institute for Material and Beam Technology (IWS), Germany; *D Hoesel,* Fraunhofer Institute for Machine Tools and Forming Technology (IWU), Germany; *V Weihnacht, A Leson,* Fraunhofer Institute for Material and Beam Technology (IWS), Germany

The superhard ta-C carbon coatings are outstandingly suited as frictionreducing protective coatings for lubricated and non-lubricated application conditions. They can be deposited on all kinds of tools and components with very good adhesion over a wide coating thickness range. The coating is applied with the Laser-Arc technology, particularly developed for ta-C coatings. In order to obtain a high ta-C coating quality with low defect densities, a plasma filtering technique is used. With hardness of up to 70 GPa, ta-C coatings nearly reach the hardness of nanocrystalline diamond coatings. This results in an unusually high resistance to abrasive wear, above all in the machining of challenging composite materials. At the same time, the carbon surface reduces adhesion of material and causes very low friction between the tool edge and the cutting chips. The combination of hardness and low friction provides ta-C coatings with a special advantage for the processing of composite materials. 2:50pm B3-5 Selection of DLC Coatings for Application in Wrist-watch Mechanisms, *Steve Franklin*, Steve Franklin Consultancy, Netherlands

Traditionally, watch manufacturers use liquid lubricants, sometimes applied to up to 100 individual lubrication points, to achieve the required low and stable friction behaviour between components and sufficient lifetime performance. Despite the careful treatment of parts and use of specific (often customized) lubricants, after a few years, the effectiveness of the lubricants degrades through evaporation, (wear) particle contamination, etc., and as a consequences watches break down or do not perform as required. In addition, some lubrication points are very difficult to access.

The goal of this study was to evaluate whether, for certain watch components, solid-lubricating coatings can be used to replace liquid lubricants.

Eight different types of Diamond Like Carbon (DLC) coatings and a titaniumstabilized MoS₂ coating (all commercially available, different suppliers), which had been preselected from a wider range of possibilities, were evaluated in laboratory tribological tests. The tests were carried out in air under reciprocating sliding conditions using an uncoated steel ball (4.5 mm diameter) sliding on a coated steel plate at 2N load, 10 mm/s sliding speed and at two levels of humidity, 30% and 70% RH. These conditions were chosen in order to simulate the actual use conditions as closely as possible. All tests were carried out three times to gain insight into the repeatability of the results.

The friction force was recorded continuously as a function of sliding distance up to 720m, corresponding to a test duration of 20 hrs. The average wear rate of the coating was determined at the end of the test, based on surface profilometry. Scanning Electron Microscopy (SEM) was used to examine the coatings and associated steel balls after testing.

Clear differences were observed in the friction and wear behaviour of the different coatings but this did not correlate well with coating hardness. Transfer layer formation from the coating onto the steel ball was observed in all cases and it is hypothesized that the formation of this layer is related to the running-in behaviour, i.e. the sliding distance needed in order to achieve a stable friction force. The characteristic appearance of the transfer layer, as observed using SEM, varied considerably between the coatings tested. Increasing the humidity from 30% to 70% had relatively small effects on the friction and wear behaviour.

Three coatings, Ti-MoS₂, a-C:H and Si-doped DLC, were found to exhibit the best performance in terms of tribological criteria specific to the application. These coatings were selected for further testing using actual watch components.

3:10pm B3-6 The Role of HIPIMS and Discharges with a Positive Voltage Reversal on Coating Properties in Industrial Applications such as Hard Coatings and DLC, *Ivan Fernandez*, *A Wennberg*, *F Papa*, Nano4energy SI, Spain; *G Eichenhofer*, HiP-V, Germany

HIPIMS (High Power Impulse Magnetron Sputtering) is a magnetron sputtering technology devoted to produce thin film coatings with enhanced properties. The technology offers advantages such as denser coatings, higher hardness values and smoother surfaces. This paper presents studies of the role the HIPIMS discharge, as well as an added positive voltage reversal pulse right after the negative HIPIMS pulse, have on coating properties and productivity in industrial applications.

The instant advantage is that the magnetron surface will be immediately discharged, which will reduce the tendency to arcing. However, there are several other effects observed during the performance of reactive sputtering, such as enhanced high energetic positive ion bombardment towards the substrate. Due to raise of the plasma potential, higher incorporation of reactive species into the depositing film, enhanced deposition rates and elastic hardness values, as well as crystallinity will be affected.

Measurements of the deposition rate, coating hardness and crystallinity have been performed for different metallic coatings (Ti, Al) as well as nitrides (TiN) and oxides (TiO2) deposited in reactive mode and more recently the effects that HIPIMS and positive voltage reversal plays on DLC coatings, in terms of adhesion as well as the functional layer.

3:30pm B3-7 Towards New Horizon for DLC Coating Technology for Automotive Components, Tetsuya Takahashi, Kobe Steel, Ltd., Japan INVITED

Minimization of energy loss due to friction of sliding parts in automotive components becomes increasingly important to increase overall energy

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efficiency of vehicle. Surface engineering through application of DLC coating is effective to lead to enhancement of wear resistance and sliding property, and hence widely used in recent automotive industry. A product range of DLC coatings in automotive components includes, for instance, fuel injection system, valve lifter, rocker arm, piston ring, gears. Items for application have been expanding more and more in recent years. Depending on its application, suitable deposition techniques shall be selected and the process parameters including design of adhesion layer are optimized accordingly. In addition to conventional hydrogen containing DLC coatings, hydrogen free DLC having a high sp3/sp2 ratio, also referred to as ta-C (tetrahedral amorphous carbon), draws a great practical attention due to its unique features of high hardness >40 GPa and a possible superlubrication effect in a particular lubricating condition. Deposition of ta-C with the controlled property and ensured adhesion is of practically significance. A productivity of coating process, i.e., throughput, is equally important especially for an industrial mass production scale.

In this work we compare systematically various DLC coatings deposited by various industrial vacuum coating technologies of, for instance, unbalanced magnetron sputtering, cathodic arc, also referred to as arc ion plating, and plasma enhanced CVD process. These are presented with respect to the coating properties, applications, and productivity. Our particular interest is also placed on ta-C coatings deposited by cathodic arc. We have developed a round-bar type target specially designed for ta-C coatings for industrial scale. The target was a pure graphite with a typical diameter of 20 mm. A stable arc discharge was sustained at a target surface either in vacuum or in inert gas atmosphere. The coatings were characterized as a low hydrogen content of <1%, high sp3 fraction of >80%, and high hardness of up to 70 GPa. Also the coatings exhibited a relatively low surface roughness in as deposited condition, implying a less emission of macroparticles without any mechanical and/or magnetic filter. For better understanding of intrinsic mechanical properties of coatings, Micro Slurry Erosion (MSE) test was performed where erosion rate of material against blasting was quantitatively evaluated and served as a representative material parameter. The results of MSE of some selected coatings are discussed in relation to the material properties and respective coating parameters.

4:10pm B3-9 DC/Pulsed Cathodic Arc Discharge for Deposition of ta-C Coatings, Xiubo Tian, P Wan, H Liu, C Gong, State Key Laboratory of Advanced Welding and Joining, Harbin Institute of Technology, China

The ta-C films have gained more interest in industrial applications due to excellent properties including good chemical stability, high hardness and wear resistance, good biocompatibility. Vacuum arc is an effective tool to deposit Ta-C films. To achieve better microstructure and surface properties, we have developed new power supply and system. The cathodic carbon arc may be driven by the specially designed power supply to produce required plasmas. With the assistance of external magnetic field, the plasmas near the samples may be optimized. In order to improve the adhesion between the film and substrate, the bottom layer and support layer were deposited. The processing parameters have to be optimized for the top ta-C coatings (thin and thick). A proper coil current (external magnetic field) was needed to achieve better adhesion of the films. A larger arc current was not favorable for better microstructure and surface properties. With the DC arc, the ta-C film possessed the nanohardness of 33.5GPa with a coil current of 0.1A. With the pulse work mode of arc power supply, a hardness of 45GPa was obtained with average current of 80A. The ta-C films have been utilized on surgical knives and cutting tools. The discharge of pulsed carbon arc, microstructure and surface properties of ta-c films will be presented.

4:30pm B3-10 A General Engineering Applicable Superlubricity: Hydrogenated Amorphous Carbon Film Containing Nano Diamond Particles, Junyan Zhang, Z Cao, State Key Laboratory of Solid Lubrication, Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, China Designing promising mechanical systems with ultra-low friction performance and establishing superlubricity regimes are desirable not only to greatly save energy but also to reduce hazardous waste emissions. However, very few macroscale superlubricity regimes for engineering applications have been reported. Here, we demonstrate that sustained superlubricity can be achieved at engineering scale when the contact pressure is higher than 2 GPa. Such engineering superlubricity originates from the in situ formation of curved graphene ribbons or onion carbon tribo-films at the sliding interface. Experimental data also demonstrate the wear of the amorphous carbon film containing some nano-diamond particles against Al_2O_3 ball is consistent with atomic attrition. A feasible two-stage mechanism is proposed to explain the friction and wear

behaviors. This finding in amorphous carbon films will not only enrich the understanding of superlubricity behavior, but also be helpful to establish more superlubricity regime for more engineering applications.

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