

## Tribology and Mechanics of Coatings and Surfaces

### Room Town & Country C - Session MC3-2-WeA

#### Tribology of Coatings and Surfaces for Industrial Applications II

**Moderators:** Osman Levent Eryilmaz, Oak Ridge National Laboratory, USA, Giovanni Ramirez, Zeiss Industrial Quality Solutions, USA

2:00pm **MC3-2-WeA-1 Effect of Electrical Current Application on the Tribological Properties of Soft and Hard ta-C Coatings on HSS Substrates**, Amir Masoud Khodadadi Behtash, University of Windsor, Canada; Woo-Jin Choi, Jongkuk Kim, Korea Institute of Materials Science, Korea (Democratic People's Republic of); Ahmet T. Alpas, University of Windsor, Canada

As electric vehicles (EVs) become more widespread, managing electrical current effects on friction and wear in moving components is crucial for enhancing durability and efficiency. Diamond-like carbon (DLC) coatings, known for their low friction and insulating properties, show potential in these applications. This study investigates the tribological characteristics of two types of tetrahedral amorphous carbon (ta-C) coatings -soft (51 GPa) and hard (69 GPa)- on high-speed steel (HSS) substrates under the electrical current application. The soft ta-C coating was deposited at 150 °C, while the hard ta-C coating was deposited at room temperature with a -100 V substrate bias, both using filtered cathodic vacuum arc (FCVA) with a Ti interlayer deposited by magnetron sputtering. The average surface roughness ( $R_a$ ) values were  $17.1 \pm 0.3$  nm for the soft ta-C coating and  $20.3 \pm 0.9$  nm for the hard ta-C coating. Friction and wear resistance were evaluated using a modified ball-on-disk tribometer with an AISI 52100 steel counterface, under electrical currents from 0 to 1500 mA. Under non-electrified conditions, both hard and soft ta-C coatings displayed low wear rates of 4.5 and  $5.27 \times 10^{-7}$  mm<sup>3</sup>/m.N, respectively. With applied electrical currents, however, notable differences emerged. The hard ta-C coating demonstrated coefficient of friction (COF) values ranging from 0.11 to 0.44 under electrical currents between 0 and 500 mA. In comparison, the soft ta-C coating exhibited lower COF values, ranging from 0.11 to 0.29, across a broader current range of up to 1500 mA. The wear rate of the hard ta-C coating increased significantly to  $1.6 \times 10^{-5}$  mm<sup>3</sup>/m.N at 300 mA, whereas the soft ta-C coating maintained a much lower wear rate of  $1.05 \times 10^{-6}$  mm<sup>3</sup>/m.N at the same current and reached only  $6.17 \times 10^{-6}$  mm<sup>3</sup>/m.N at 1200 mA. These results indicate that the electrical current carrying tribological performance of ta-C coatings on HSS substrates can be tailored by heat treatment to enhance their response. Raman spectroscopy and electron microscopy are utilized to delineate the mechanisms underlying these structural changes and will be presented at the conference.

2:20pm **MC3-2-WeA-2 Impact of Electrification on the Tribological Performance of Metal Doped a-C Coatings**, Miguel Rubira Danelon, Newton Kiyoshi Fukumasu, Roberto Martins de Souza, André Paulo Tschiptschin, University of São Paulo, Brazil

Amorphous carbon (a-C) coatings, composed of sp<sup>2</sup> and sp<sup>3</sup> hybridizations of carbon, may enhance the surface properties of materials. These coatings are commonly used as solid lubricants, improving tribological performance by forming a tribolayer that reduces the coefficient of friction by graphitization. In many systems, a-C coatings offer the potential to lower frictional energy losses and wear, improving efficiency and durability. Specific phenomena are anticipated for electric vehicles (EVs), since, from one side, electric current can affect surface wear in electrified systems by promoting accelerated oxidation or arc formation. On the other hand, electrical current flowing through an a-C coated contact can induce carbon crystallization, benefiting EV engine performance. Pure a-C lacks the conductivity needed for this crystallization effect, which can be improved by doping the a-C with metallic elements. Using copper or nickel as dopants can reduce electrical resistivity and catalyze carbon nanostructure formation, further reducing friction. This study investigates the tribological behavior of metal-doped a-C coatings under electrified ball-on-plane tests. Me:a-C coatings were deposited on glass substrates using pulsed DC balanced magnetron sputtering. Ni and Cu were used as dopants, with different concentrations, to improve electrical conductivity. Tribological tests involved a ball-on-plane setup with a 10 N normal load, 5 mm stroke, and 0.28 Hz frequency, applying 30 V in four current flow modes: current flowing from ball to plane, from plane to ball, no current, and intermittent on-off cycling every minute. The coatings' microstructure and composition were analyzed using Scanning Electron Microscopy with Energy-dispersive X-ray spectroscopy (EDS). Raman spectroscopy was used to evaluate carbon

structure, while instrumented indentation tests allowed the characterization of mechanical properties. Results showed that doping a-C is essential to promote a direct response to electrical stimulation. Increasing the metal content of the amorphous-carbon coating increases the conductivity but decreases the wear resistance, due to a higher metal content. In contrast, reducing the metal content leads to insufficient conductivity, hindering the electrical current's effect on carbon graphitization. Current flow promoted friction coefficient variations, which were not influenced by thermal effect, since no significant temperature increase was observed. Instead, COF variations were related to instant changes in current flow during contact. The wear resistance has also been influenced by the current, with different outcomes depending on the current direction.

2:40pm **MC3-2-WeA-3 Graphene-Related Materials: Bridging Fundamental Tribology and Industrial Applications Across Multifarious Environments**, Mingi Choi, Ji-Woong Jang, Pusan National University, Republic of Korea; Anirudha Sumant, Argonne National Laboratory, USA, India; Ivan Vlassiouk, Oak Ridge National Laboratory, USA, Russian Federation; Jae-Il Kim, Korea Institute of Materials Science, Republic of Korea; Young-Jun Jang, Korea Institute of Material Science, Republic of Korea; Songkil Kim, Pusan National University, Republic of Korea

Solid lubricants play a crucial role as alternatives to liquid lubricants in extreme environments and as solutions for enhancing mechanical system performance under ambient conditions at the macroscale. Among these, graphene, a representative two-dimensional nanomaterial, has attracted significant attention due to its exceptional nanoscale tribological properties. However, its application as a solid lubricant for macroscale industrial systems remains a challenge. Recent studies have highlighted that tailoring graphene's properties through functionalization, oxidation, can significantly enhance its performance. This underscores the strong correlation between the tribological behavior of graphene-based materials and their elemental and compositional properties.

In this work, we demonstrate the versatility of graphene-related materials as solid lubricants by engineering their structural and compositional properties. Under ambient conditions, we developed a heterogeneous structure of graphene oxide layered on pristine graphene, achieving over 100 times greater durability (>10 km) compared to pristine graphene (~100 m) while maintaining its low COF. In contrast, under humidity- and oxygen-free environments, pure graphene oxide exhibited a super low coefficient of friction (COF). Remarkably, in an argon environment, the COF approached the superlubric regime (COF < 0.01), while in vacuum, the COF gradually increased to 0.07. By unveiling the intrinsic lubrication mechanisms of graphene oxide in these environments, we highlight the potential of graphene-based materials as solid lubricants for diverse engineering applications, bridging fundamental understanding with industrial relevance.

3:00pm **MC3-2-WeA-4 Evaluation of the Reduced Bearing Wear Through Plasma Nitriding for Use in Wind Turbines**, Arthur Cid de Abreu, Rayane Dantas da Cunha, João Freire de Medeiros Neto, Salete Martins Alves, Federal University of Rio Grande do Norte, Brazil

In 2023, significant advancements in the wind industry led to a record-breaking achievement: over 100 GW of new onshore wind installations and the second-largest total for offshore wind at 11 GW. This type of energy production has proven profitable for investors as it represents a viable source of green and sustainable energy. The trend indicates an expansion of wind farms in the world over the coming years. However, a significant concern in this sector is the damage caused by operational characteristics, such as dynamic loading and severe operating conditions. These factors can lead to reduced efficiency and increased maintenance costs. To address this, developing surface treatments with high wear resistance and friction reduction capabilities is essential to ensure the longevity and efficiency of mechanical components. The main goal of this research was to extend the service life of wind turbine components through plasma nitriding surface treatments to enhance their wear resistance. The study consisted of carrying out a plasma nitriding process in bearing balls of steel AISI 52100 under different temperatures and treatment times to improve wear resistance. Tribological tests were performed to evaluate the wear resistance of the bearings using a pin-on-disc tribometer with non-conformal contact (ball-disc). The disc rotated at a speed of 500 rpm, with an applied load of 60 N for 30 minutes. Tests were performed both in dry and lubricated conditions. After the tribological tests, the discs and balls were analyzed by optical microscopy and 3D and 2D profilometry analysis to quantify the worn volume. The wear mechanisms and morphology were

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further examined using scanning electron microscopy and energy-dispersive spectroscopy (EDS). The results indicated that the plasma nitriding treatment applied to the SAE 52100 steel balls significantly improved the surface tribological properties, characterized by a reduction in the friction coefficient and decreased wear on the SAE 1045 steel discs.

**3:20pm MC3-2-WeA-5 Plasma Nitriding of Quartz, Stephen Muhl**, Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México; **Julio Cruz, Marco Martinez**, Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México

Plasma nitriding is a valuable and well-established technique for surface hardening of metals to improve their mechanical and tribological properties, such as hardness and wear resistance. Typically, plasma nitriding involves the use of a glow discharge of a mixture of nitrogen and hydrogen, where the metal component to be treated is the cathode and the chamber wall is the anode. The low-pressure plasma (15–1500 Pa) produced by the application of a DC potential (0.3–1.0 kV) contains nitrogen ions, which are accelerated towards the cathode and implanted in the surface of the metal. The treatment time, surface concentration of nitrogen, and temperature of the metal component determine the depth and gradient of the nitride layer, but various tens of microns are often formed. The same process cannot directly be used to nitride insulators since such materials cannot be used as a cathode in a DC plasma.

We have developed a variant of the normal plasma nitriding scheme where the discharge is produced by applying an RF potential and the piece to be nitrided is mounted on a magnetron cathode. This is, of course, the same as a RF magnetron sputtering system. Previous studies have shown that the DC voltage bias generated on the surface of an insulating target used in a RF magnetron sputtering cathode depends on the following factors: the relative areas of the anode and cathode, the applied RF power, the gas pressure and composition, and the degree of matching of the impedance of the electrical supply to the impedance of the plasma. We have measured the DC bias potential and the rate of sputtering of a quartz target mounted on a 2" MAK magnetron cathode in a pure nitrogen gas discharge as a function of the area of the anode, the gas pressure, the applied power, and the degree of matching indicated by the ratio RF(Reflected Power) / RF(Forward Power). Using conditions which produced a minimum sputter etching of the target, we produced three samples nitrided for 60, 90 and 120 min. We present the thickness and composition of the nitride layer measured using XPS and RBS, and the hardness and wear resistance of these layers.

**3:40pm MC3-2-WeA-6 Penetrability: A New Parameter for Wear Estimation of Multilayer Coatings, Muhammad Usman**, City University of Hong Kong

Many efforts are being made to predict the wear of thin films/coatings through indentation. The 10%-depth-hardness defined by the ISO 14577-4 standard to avoid the substrate effect is commonly used for wear estimation of single-layer coatings. The coating structure varies from layer to layer (top to bottom) in multilayer coatings, and no clear guidelines for hardness measurement are available for them at the time of writing. Moreover, hardness is a system parameter that may change with depth, indenter tip shape and size, micro to nanofilm thickness, and the substrate effect. Therefore, this method may not be adequate for multilayers, multiscale nanostructured materials, and composites. To address the issue, we hypothesize that for the same material (in our case, various C/C multilayer coatings), there should exist a correlation between the mechanical work done due to wear and the mechanical work done due to indentation. A thorough experimental investigation was conducted using a range of multilayer diamond-like carbon coatings at different loading conditions to verify our hypothesis. The penetrability (newly proposed parameter) correlates with the wear process. The test drive of the method successfully predicted multilayer coatings in terms of wear behavior trends and identified coating designs with comparatively low and high specific wear rates. It is anticipated that with further investigations employing penetrability to thin film wear research, the approach could be used to understand the wear of thin films in a more universal manner.

**4:00pm MC3-2-WeA-7 Structure and Tribo-Mechanical Properties of Si-Containing Ta-C Thin Films Grown by Cathodic Arc Evaporation, Nelson Filipe Lopes Dias, Julia Urbanczyk**, TU Dortmund University, Germany; **Domic Stangier**, Oerlikon Balzers Coating Germany GmbH, Germany; **Jörg Debus, Wolfgang Tillmann**, TU Dortmund University, Germany

Among various types of diamond-like carbon, tetrahedral amorphous carbon (ta-C) thin films have attracted considerable attention due to their high hardness of up to 70 GPa, low friction, and high wear resistance. This

property profile makes ta-C a particularly promising thin film system with broader application potential compared to the well-established hydrogen-free amorphous carbon (a-C) and hydrogenated amorphous carbon (a-C:H). For both a-C and a-C:H, thin film properties are typically tailored by incorporating modification elements to meet specific application requirements. In this context, silicon (Si) is widely used to improve the thermal stability and reduce friction under dry sliding conditions. As a result, the modification of ta-C by Si lies within the focus of recent research to tailor its thin film properties.

A key challenge in the synthesis of ta-C:Si is the precise incorporation of low Si concentrations into the thin film without significantly reducing the high fraction of sp<sup>3</sup>-coordinated carbon (C) bonds, which would compromise the superior hardness of ta-C. To overcome this, Si-containing graphite with 2.5 and 5 at.% Si as well as pure graphite were used as cathode materials and were mounted on an array of cathodic arc evaporators arranged vertically. By positioning AISI M2 steel substrates at different heights in front of the evaporators, Si-containing ta-C thin films with low Si concentrations were successfully deposited.

This vertical cathode arrangement allows for tailoring the Si content gradually decreases from top to bottom and for reaching a Si-free ta-C thin film at the lowest height. The hardness of ta-C:Si decreases with increasing Si content but remains above 40 GPa even at the highest Si concentrations. Notably, a high hardness exceeding 60 GPa is achieved at the lowest Si content. To correlate the mechanical properties with the structural characteristics, Raman spectroscopy with UV laser excitation was performed for precise structural analysis of the sp<sup>3</sup>-coordinated C bonds. Additionally, tribometer tests were conducted to evaluate the influence of Si content on the friction and wear behavior at room temperature. The results highlight the potential of depositing low Si-containing ta-C:Si thin films with superior tribo-mechanical properties using cathodic arc evaporation and low Si-containing graphite cathode materials.

**4:20pm MC3-2-WeA-8 Recent Advances and Strategies for High-Performance Coatings Deposited by the Advanced Magnetron Sputtering, Yixiang Ou**, Beijing Academy of Science and Technology, China

Coatings with micro- or nano-scaled structure are always fabricated by various techniques to fulfill the requirements of unique performances according to special working conditions. The differences in preparation techniques always result in large differences in their properties, even for the same coating materials. High-performance coatings are solely up to surface integrity, morphology, microstructure, stress state, interfacial structure, cohesion and adhesion that are all strongly affected by preparation processes. Therefore, how to take advantage of the numerous deposition techniques to design a right preparation process to produce coatings of high performance to meet the demands is the real question. The combined magnetron sputtering (HiPIMS, DOMS and PDCMS) exhibit a good advantage in the preparation of functional hard coatings. Hence, we summarized designs, preparation and application of typical high-performance coatings, such as flexible hard coatings, superhard yet tough coatings deposited by the combined magnetron sputtering.

**Keywords:** High-performance coatings; Functional hard coatings; Superhard yet tough coatings

**4:40pm MC3-2-WeA-9 Breaking the Hardness-Toughness Dilemma in Protective Coatings: Controllable Preparation and Mechanism Exploration of Superhard yet Tough Coatings, Haoqi Wang**, Beijing Academy of Science and Technology, China

Superhard coatings have been widely applied for surface protection of components. However, due to the intrinsic trade-off between hardness and toughness in surface engineering, most conventional superhard coatings achieve their exceptional hardness at the expense of compromised toughness. Low-toughness hard coatings are prone to brittle fracture during service life, leading to premature failure via microcracking propagation. This study addresses this challenge by developing superhard and tough surface coatings through high-intensity pulsed magnetron sputtering (HIPIMS). An innovative approach combining 3D layer-by-layer scanning reconstruction with high-resolution transmission electron microscopy (HRTEM) characterization enables multiscale visualization of indentation-induced microcrack networks in the coatings. By systematically analyzing geometric features, size distributions, and spatial structural relationships of microcracks across nanoscale to micrometer scales, new insights into the toughening mechanisms of superhard coatings are revealed. This research provides a novel pathway for advancing the development of high-performance protective coatings with balanced hardness and toughness.

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**Keywords:** Superhard yet tough coatings; HiPIMS; Visualization of cracks

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