## Surface Engineering - Applied Research and Industrial Applications
## Room Town & Country C - Session IA2-1-TuM
## Surface Modification of Components in Automotive, Aerospace and Manufacturing Applications I

**Moderator: Jan-Ole Achenbach**, KCS Europe GmbH, Germany

8:00am **IA2-1-TuM-1 Influence of Plasma Carburizing on Corrosion Behavior and Interfacial Contact Resistance of Austenitic Stainless Steels***, **Phillip Marvin Reinders (p.reinders@tu-braunschweig.de)**, P. Kaestner, G. Bräuer,* Technische Universität Braunschweig, Germany

Austenitic steels are known for their high corrosion resistance but at the same time have low wear resistance and high interfacial contact resistance (ICR), which limits their application e. g. as bipolar plates in Proton Exchange Membrane Fuel Cells (PEMFC). Plasma diffusion treatment, specially the well-known plasma nitriding, improves the hardness and interfacial contact resistance but mostly worse the corrosion behavior in PEMFC environment.

Aim of this study is to evaluate the less known plasma carburizing as a suitable process for functionalization austenitic stainless steels. For this purpose, a number of processes were executed under specific variation of temperature ranging from 360 °C to 450 °C and duration of 10 to 16 h. The samples were analyzed using x-ray diffractometer, x-ray photoelectron spectroscopy, SEM, Vickers microindentation, potentiodynamic polarization and ICR measurements.

It could be shown that the corrosion current density (1.78 $\mu A \cdot cm^{-2}$) of the treated samples are an order of magnitude lower than those of the reference (17.38 $\mu A \cdot cm^{-2}$). The ICR was also reduced from > 1000 $m\Omega \cdot cm^{-2}$ down to 31 $m\Omega \cdot cm^{-2}$. After corrosion, even lower values around 15 $m\Omega \cdot cm^{-2}$ were achieved. The targets according to DOE ( < 1 $\mu A \cdot cm^{-2}$ and < 10 $m\Omega \cdot cm^{-2}$) were almost achieved. A comparison to the plasma nitrided samples was also performed and shows the high potential of plasma carburizing.

Keywords: plasma carburizing, s-phase, austenitic stainless steel, corrosion behavior, interfacial contact resistance, bipolar plate

8:40am **IA2-1-TuM-3 Tribological and Corrosion Behaviour of Crn and AlCrn Coatings over Nitrided Medium Alloy Steel***, J. Maskavizan, E. Dalibon,* National University of Technology (UTN), Argentina***; Sonia Brühl (sonia@frcu.utn.edu.ar)**,* National UNiversity of Technology (UTN), Argentina

Different Cr based coatings were deposited over medium alloy AISI 4140 steel in industrial facilities (Oerlikon Balzers Argentina), to improve wear and corrosion resistance in aggressive environments, like the plastic forming industry, and other applications in the aluminum industry. As AISI 4140 is a soft substrate, tests were carried out in two conditions: i) quenched and tempered (Q&T), ii) Q&T plus ion nitriding.

Friction and adhesive wear test were in a carried out in a rotational pin on disk machine using an alumina ball 6 mm in diameter as counterpart. The coatings were characterized by SEM and XRD. The corrosion test consisted in anodic polarization in a chloride solution. Finally, the film adhesion was tested by Rockwell C indentation and Scratch test at constant loads.

Both coatings resulted about 2.7-3 microns width. They presented good adhesion tested with Rockwell C indentation over nitrided substrates but not so good (HF3) for unnitrided ones. In the scratch test the critical load was over 50 N for the CrN but the AlCrN presented some spallation at the same load.

The CrN coatings presented the lower coefficient of friction in the Pin on Disk test at 5 N load. To measure wear loss, 12 N was used in the duplex case, meaning nitrided plus coating. The wear volume was less in the CrN too. In the corrosion test, only the CrN film showed a quasi-passive zone in NaCl solution, meanwhile the AlCrN presented active dissolution.

The observation of the wear tracks and the film microstructure, so as the surface after corrosion, allowed to explain the difference between nitrided and non nitrided substrates primarily, having this last combination a low load bearing capacity. Between both films, some slightly differences between mechanical properties explain the best behavior of CrN.

9:00am **IA2-1-TuM-4 Influence of the Cathodic Bias Parameters on Corrosion Resistance in the Micro-Arc Oxidation Coating of AZ31B Magnesium Alloy***, **Shih-Yen Huang (f08525129@g.ntu.edu.tw)**, Y. Lee, Y. Chu,* National Taiwan University, Taiwan

Micro-arc oxidation (MAO) is a surface treatment applied to valve material to form a multifunctional ceramic coating based on the principle of anodizing. By regulating electrical parameters and adjusting electrolyte composition, the MAO coating has the capability to meet diverse specifications across numerous domains. Among the various MAO process equipment, the bipolar pulse power supply stands out for its flexible process parameters and fast coating growth rate, which is attributed to the introduction of cathodic bias. The incorporation of cathodic bias has been proven to benefit the properties of the MAO coating by reducing the discharging energy and promoting the crystalline transition within the oxide phase of aluminum. However, the impact of cathodic bias in the MAO process is seldom discussed in magnesium alloy applications.

In this research, AZ31B magnesium alloy was used as the substrate to produce MAO coating, with the objective of clarifying the mechanism of cathodic biason the growth mechanism of MAO.Under controlled anodic bias parameters and cathodic duty ratio,the best corrosion resistance, as observed in the electrochemical impedance spectroscopy (EIS) result, was achieved with an impedance value of $2.55 \times 10^6\ \Omega \cdot cm^2$ when the total charge quantity input through cathodic bias equaled that through anodic bias.Under the same condition, the corrosion resistance decreases regardless of whether the cathodic charge quantity is higher or lower than the anodic charge quantity, and a significant decrease in impedance value by two orders of magnitude was found when the ratio of cathodic charge quantity to anodic charge quantity exceeded 1.33. Moreover, under controlled cathodic charge quantity, MAO coatings were found to exhibit an impedance value of $10^6\ \Omega \cdot cm^2$ while the ratio of cathodic current density to anodic current density remained below 1. However, there was a notable decline in impedance value when the ratio exceeded 1.33.These results suggest that the influence of both the total charge quantity and the instantaneous input current density of the cathodic bias on the corrosion resistance of MAO coatings might be attributed to the limiting current density in the cathodic bias period.

9:20am **IA2-1-TuM-5 Nanolubricants: Pioneering Sustainable Solutions for the Lubrication Industry***, **Anirudha Sumant (sumant@anl.gov)**,* Argonne National Laboratory, USA **INVITED**

Over the past decade, the forefront of tribological studies has been illuminated by the exceptional properties of graphene, along with other 2D materials and their synergies with various nanomaterials. These cutting-edge nanolubricants have demonstrated unparalleled wear and friction performance across diverse systems. Their remarkable ability to achieve near-zero levels of friction and wear (known as superlubricity), extends even to macroscopic scales in different environments and under moderate to high contact pressures. This positions them as a promising alternative to traditional oil-based lubricants. Despite their impressive performance, the sustained and long-term reliability of these solid nanolubricants under more intricate tribological conditions remains a subject of ongoing investigation. Establishing their credibility as a potential replacement for oil-based lubricants necessitates a deeper understanding of their behavior in complex scenarios.

At Argonne National Laboratory, we have made significant strides in developing various nanolubricants. Our research showcases the attainment of superlubricity on rough steel contacts, even under high contact pressures (~1GPa), in both linear and sliding-rolling contacts as well as at high temperatures in an ambient environment. Furthermore, these nanolubricants exhibit stability over extended periods, enduring 70 kilometers of linear sliding without failure.

Our investigation delves into the role of tribochemistry at the micro/nanoscale and its profound impact on tribological performance at the macroscale. We present compelling examples resulting from collaborations with industry partners, particularly within the automotive sector, focusing on applications such as metal stamping. This progress not only sets the stage for future breakthroughs but also marks a significant stride toward realizing oil-free superlubricity in real-world applications. By doing so, these research efforts make a substantial contribution to the broader mission of decarbonization and offer sustainable solutions for the evolving lubrication industry

10:00am **IA2-1-TuM-7 Structural – Tribological Performance Evaluation of Ti-6Al-4V ELI Alloy after Sequential Surface Treatments***, **Daniel Toboła (daniel.tobola@kit.lukasiewicz.gov.pl)**, P. Chandran,* Łukasiewicz Research Network – Krakow Institute of Technology, Poland*; J. Morgiel,* Institute of Metallurgy and Materials Science of Polish Academy of Sciences, Poland

Titanium alloys are characterized by high specific strength, formability and corrosion resistance, but poor wear. The high cost of both metallurgical processing of its ore as well as later mechanical working, machining and the need to improve surface hardness by a proper treatment generally limits its wider application to aviation or military industry. The durability of the mechanically or physico-chemically upgraded surface layer depends to the largest extent on its: (i) geometrical irregularities and (ii) microstructure changes in the sub-surface area, which decide on the surface integrity of the material. Optimizing the latter is critical for components being employed in sliding or rolling contact with other parts and are subject to rapid wear resulting in significantly reduced life-times [1]. Hence, this study focuses on the influence of plastic deformation of near surface areas induced by slide burnishing/shot peening followed by sequential gas and plasma nitriding processes on the tribo-mechanical properties of the Ti-6Al-4V ELI alloy.

The Ti-6Al-4V ELI alloy substrates were subjected to heat treatment followed by a sequence of surface treatments like: turning (T), turning + burnishing (T+B), turning + burnishing + gas/plasma nitriding (T+B+GN/PN), turning + shot peening (T+SP), turning + shot peening + gas/plasma nitriding (T+B+GN/PN). Details of the mechanical treatments and gas nitriding parameters were described in our previous work [2,3]. Subsequently, all the substrates modified in this way were subjected to a detailed investigation involving both their phase composition and microstructure characterization as well as assessment of friction coefficient, hardness and sliding wear properties. Preliminary investigations revealed the in-situ formation of a very thin amorphous 'tribo-layer' which could prove to be beneficial during tribological contact. The effect of surface mechanical working applied as pre-treatment for the gas and plasma nitriding on the enhancement of tribo-mechanical properties of Ti-6Al-4V ELI alloy will be discussed in detail in the presentation.

1. Philip JT. et al., *Friction*, 7(6), (2019) 497–536
2. Toboła D. et al., *Appl. Surf. Sci.*, 515 (2020) 145942
3. Toboła D. et al., *Appl. Surf. Sci.*, 602 (2022) 154327

10:20am **IA2-1-TuM-8 Wear Particle Emission Influenced by Surface Conditions of an Alumina-Coated Cast Iron Disc***, **Ran Cai (cai12r@uwindsor.ca)**, X. Nie,* University of Windsor, Canada*; Y. Lyu,* Lund University, Sweden

Hard coatings can be applied to a traditional cast iron brake disc to increase wear resistance and thus reduce brake particle emission. An alumina coating prepared through a modified plasma electrolytic oxidation (PEO) process also shows a promise in wear reduction for the automotive brake disc. This work was to study effect of the alumina coating on the wear particle emission of the cast iron brake disc using a dedicated pin-on-disc (PoD) tribotester combined with an airborne particle emission measurement system. The testing samples included uncoated and alumina-coated cast iron discs with different surface roughness finish. Two different commercially available brake pad materials—non-asbestos organic (NAO) and low-metallic (LM)—were used as tribotesting counterparts. To simulate surface roughening effect by de-iced salt corrosion, salty water was sprayed on the sample surface and let it dry between the interval of subsequent tribotests. The data evaluation covered coefficients of friction (COF), specific wear rates, particle number concentrations, and particle size distribution. The friction tribolayers and emitted particles were analysed using scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX) for better understanding of morphology and elemental compositions of the particulates. More discussions were given to the PEO coating process in terms of its role played in coating surface texturing, tribolayer formation, and wear particle emission at the disc surface.

10:40am **IA2-1-TuM-9 Metal Coated Carbon Fiber EMI Shielding Material***, Y. Li,* National United University, Taiwan*; H. Chen,* Michigan State University, USA*; S. Chen,* National Yang Ming Chiao Tung University, Taiwan*; S. Chen, Z. Hsieh, **Chien-Chon Chen (ccchen@nuu.edu.tw)**,* National United University, Taiwan

An electroplating method was used to modify the surface morphology of carbon fiber bundles. It deposits a layer of nickel film on the surface of carbon fiber bundles. The process can further apply to the subsequent applications of carbon fiber in electromagnetic wave shielding materials and metallurgical bonding at the interface of metal-based composites. In this study, the carbon fiber bundles' surface was first treated with hot nitric acid (80 °C, 30 min) to remove the polymer and activate the carbon fiber surface. Subsequently, a direct current electroplating method (3.5 V, 30 min) was used to deposit a 2 μm thick nickel film on the surface of carbon fiber bundles and carbon fiber fabric with a diameter of 7 μm. The weight of the carbon fiber increased by 1.9 times after nickel electroplating on the carbon fiber surface. To reduce the weight of the final product, efforts can be made in the future to decrease the thickness of the nickel layer. However, it is important to consider that as the nickel layer thickness decreases, the coverage of the nickel layer on the carbon fiber surface will also decrease.This research paper also provides detailed research and discussion on the relevant processes of metallization treatment on carbon fiber surfaces, including the design of electroplating fixtures, surface pre-treatment, electroplating treatment, and post-treatment.In this paper, hot nitric acid is used to replace high-temperature decomposition of polymer on the carbon fiber surface. Electroplating is employed to deposit nickel metal on the surface of carbon fiber bundles, with control over the voltage and duration of the electroplating process. The obtained nickel film thickness is observed, measured, and analyzed. Furthermore, the electromagnetic wave shielding properties of the carbon fiber with nickel electroplating are measured.

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