Surface Engineering - Applied Research and Industrial Applications
Room Town & Country C - Session IA1-MoA

Advances in Application Driven Research and Hybrid Systems, Processes and Coatings

Moderators: Ladislav Bardos, Uppsala University, Sweden, **Vikram Bedekar**, Timken Company, USA, **Hana Barankova**, Uppsala University, Sweden

1:40pm **IA1-MoA-1 PVD Thin Film Coating Materials in Semiconductors and Impact of CHIPS Act***, Shlok Sundaresh (sshlok91@gmail.com),* Tosoh SMD, Inc., USA **INVITED**

The CHIPS and Science Act in the United States has led to significant investments in domestic semiconductor manufacturing recently. It details the importance of building a resilient domestic supply chain with funding emphasis on construction, expansion, or modernization of commercial facilities. Semiconductor manufacturing involves numerous processing steps and one of those critical steps is thin film deposition of materials on wafers to form various patterns using PVD technology. Continued pursuit of Moore's law warrants advances in technology, and materials innovation plays a key role for achieving this. PVD sputtering target material developments are extremely critical for the performance of semiconductors as these are used as consumable sources for building them. The CHIPS Act has recognized this providing specific examples of PVD sputtering targets. The talk will thus focus on advances in manufacturing of key materials for sputtering these thin films in semiconductors along with the potential boost from the CHIPS Act towards this technology.

2:20pm **IA1-MoA-3 Production and Characterization of Coating-Substrate Combinations for Ceramic Data Storage Media***, Erwin Peck (erwin.peck@tuwien.ac.at),* TU Wien, Institute of Materials Science and Technology, Austria*; B. Hajas,* TU Wien, Austria*; A. Kirnbauer, L. Kreuziger,* TU Wien, Institute of Materials Science and Technology, Austria*; C. Pflaum,* Ceramic data solutions holding GmbH, Germany*; G. Liedl,* TU Wien, Austria*; P. Mayrhofer,* TU Wien, Institute of Materials Science and Technology, Austria

Nowadays data storage and its sustainability is a topic of great importance, not only for cloud providers but also for other companies and even for people in their personal lives. Most of the data stored is referred to as cold data, meaning it is very rarely changed and accessed (e.g. photos, research results). That cold data must be stored, in order to do that, cloud providers run server farms utilizing hard drive discs (HDD). In that way they make the data available on the users' demand. Those server farms need a lot of energy, and the storage capacity is limited. To overcome the issue of needed energy and limited capacity, a new form of storage media is in the focus of our research. By utilizing a certain coating-substrate combination, it is possible to write data into ceramic data carriers using a femtosecond laser. By applying this method, it is possible to write a large amount (1.25 Gigabyte) of data onto a relatively small area (100 cm^2) of the ceramic data carrier. Within our research we analyzed different coating-substrate combinations regarding their mechanical properties and laser ablation characteristics. The coatings investigated were synthesized by magnetron sputtering and argon nitrogen gas mixture using different composite targets e.g. Ti, Cr, TiAlCr, and AlCrNbTaTi. The coatings were deposited on different substrates including sapphire, silicon, glass, and austenitic steel. All the coatings were investigated by XRD showing a single-phase fcc-structure and hardness values ranging from 21 to 33 GPa. After investigating structure and mechanical properties, laser ablation tests were conducted to determine the laser ablation threshold and to find suitable coating-substrate combinations for the aimed application. Furthermore, after writing data into the samples, the samples were tested for their thermal stability, oxidation resistance, and corrosion resistance. These studies prove the exceptional stability and durability of such ceramic data storage media. Once written, storing the data is almost without any energy consumption and such ceramic data carriers would allow to save 99% of the currently used energy for storing such data.

2:40pm **IA1-MoA-4 Microstructure Tuning of MXene ($Ti_3C_2T_x$) Systems for Device Applications***, Sangeeta Kale (sangeetakale2004@gmail.com), S. Kale, D. Sable,* Defence Institute of Advanced Technology, India **INVITED**

Titanium Carbide (Ti_3C_2Tx, MXene)materials, which are obtained via systematic removal of Aluminium (Al) layers from Ti_3AlC_2 (MAXene) system, have caught extreme due to their interesting structure of alternating two-edge shared octahedral layers of Ti_6C and highly porous accordion-like structure [1-2]. MXene shows work-function tuneability, porosity variations and varied surface-chemistry interplay, as a function of different synthesis processes [3]. Sensors, Schottky diodes, energy harvesters, and storage devices are envisaged from these materials [4].

Removal of Al layers using hydrofluoric acid (HF) is one common approach to convert MAXene to MXene and create porous structures and active surface states between the inter-digited octahedra structures. On the other hand, along with various other physical processes, pulsed laser deposition (PLD) system yield a range of thin films from stoichiometric high-quality thin films to defect-engineered films.

This talk will explore three different studies: i) bulk studies on the HF-etched-Ti_3AlC_2, yielding a tuneable work-function system, as a function of acid concentration [3] ii) thin films of Carbon-deficient Ti_3AlC_2 using PLD showing semiconducting behaviour on n-Si substrate [4]; and iii) bulk chemical treatment of MXene- molybdenum oxide (MoO_3) [5] nanocomposites to form a mutually synergistic system for gas (NH_3) sensing at room temperature. Ti_3AlC_2 material show p-type behavior; when deposited on n-Si or Alumina substrate, with strained growth depending upon the substrate; with different termination groups and morphological differences. Chemically synthesized MoO_3-MXenenanocomposites evolve as a synergistic system with improved room-temperature sensing sensitivity of MoO_3 along with a stable, yet highly reactive -O, -OH and -F sites of MXene surface. These studies are further explored for wide range of device applications.

References

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3:20pm **IA1-MoA-6 Decorative Coatings in Watch Making Industry: From Laboratory to Industry***, Joël Matthey (joel.matthey@positivecoating.ch),* Positive Coating SA, Switzerland*; O. Banakh,* Haute Ecole Arc Ingénierie, Switzerland*; L. Steinmann,* Positive Coating SA, Switzerland

Discovered in the late 1960's, the atomic layer deposition (ALD) is nowadays an established and widespread technology implemented in the industry. Despite being still predominantly applied to semiconductor devices, ALD has recently found its path into new sectors. One of them is the watchmaking niche market where design and reliability play a major role in luxury products. Due to its unique features, ALD offers attractive decorative coatings on complex components and brings innovation in terms of corrosion barrier. It is especially valid when combining the benefits of ALD with other technologies such as magnetron sputtering or electroplating. The aim of this presentation is recounting the extremely fast technology transfer of ALD from laboratory experiments to industrial scale processes. Through results and achievements, the fruitful collaboration between the University of Applied Sciences (HE-ARC) and Positive Coating SA is presented. Throughout the manufacturing sequence, the demanding requirements to obtain high-quality decorative coatings are discussed. When operating ALD technology to color tridimensional parts, simulated and experimental results show that fluidics regularly prevails over ALD process parameters. Furthermore, innovative processes using ALD as a substitute for obsolete technologies are addressed: namely red-gold anti-tarnishing, brass corrosion protection, and two-colored process without masking. Despite successful accomplishments, the technical and industrial challenges to tackle in the coming years are listed to evolve the ALD technology from the semiconductor to the decorative world. The conclusion is illustrated by specimens of luxury watches where decorative coatings highlight superb designs.

4:00pm **IA1-MoA-8 Real-Time Particle Detection for Enhanced Coating Deposition Processes***, Sylvain LeCoultre (sylvain.lecoultre@bfh.ch), C. Rieille,* Berner Fachhochschule ALPS, Switzerland **INVITED**

Coatings and the associated vacuum deposition processes will play an increasingly significant role in upcoming technological trends, particularly in the fields of photonics, optics, and Industry 4.0. However, the demands for these applications are imposing increasingly stringent requirements in terms of defect size and particle inclusions within functional layers. This is primarily attributed to the ongoing reduction in the size of device structures. Particles ranging in size from a few hundred nanometers to a few microns have proven to be a major challenge during various deposition processes. These minuscule particles often lead to component failures, resulting in unacceptably high rejection rates. Therefore, the development of deposition technologies capable of monitoring and significantly reducing the incorporation of particles into coatings is essential to access and succeed in these emerging markets.

As part of a multi-partner research project, we are focusing on the development of methods for the detection and real-time monitoring of particles generated in physical vapor deposition (PVD) processes, with particular emphasis on electron beam deposition and sputtering systems. Our research objectives include understanding the different sources of particle generation, whether related to the process, mechanical movements or the cleanliness of the deposition reactor during a production campaign. It also involves determining their size distribution and tracking their velocity in the vacuum environment with spatial and temporal resolution. In addition, we aim to contribute to the development of applicable strategies for eliminating particle sources during the vacuum deposition process, thereby increasing production yields.

To achieve these goals, we are engaged in the research and development of an in situ particle detector solution based on the fundamental principles of visible light beam scattering by particles. The chosen method will be compared with other possible particle detection methods suitable for high vacuum environment. First results on particle detection during different phases of a deposition batch will be presented. In addition, a first insight into the development of a data analysis algorithm that could enable informed decisions to be made for the maintenance of parts to be changed will be discussed.

4:40pm **IA1-MoA-10 Microscopic Characterization of Optical Properties and Film Thickness Using Imaging Spectroscopic Ellipsometry***, H. Noh,* ***Alejandro Ponilla (alejandro@parksystems.com)****,* Park Systems, USA

Ellipsometry is a well-known, non-destructive optical method to measure a thin film's thickness and optical properties. It has been widely used to characterize the complex refractive indices of materials or to control the quality of a film's thickness in manufacturing processes. Demands on microscopic characterizations of optical properties have been greatly increased for new materials and structures such as 2D materials, photonic devices, to name a few. Conventional ellipsometry, however, has been restricted to a spatial resolution of several tens of microns due to the spot size limitation. Here, we introduce imaging spectroscopic ellipsometry (ISE), which enables 1-micron lateral resolution, and its application to novel materials and structures. The ISE technique can be extensively used for new materials research and quality control of industrial applications.

5:00pm **IA1-MoA-11 Plasma PVD by Small Spiral Ta Hollow Cathode***, H. Baránková, N. Suntornwipat,* ***Ladislav Bardos (ladislav.bardos@angstrom.uu.se)****,* Uppsala University, Angstrom Laboratory, Sweden

Small spiral hollow cathodes represent interesting options for local plasma processing applications. The radio frequency powered small diameter spiral hollow cathodes made from 0.45 mm diameter Ta wire rolled around 0.5 mm diameter rod have been tested in coatings by physical vapor deposition (PVD) on silicon substrates at gas pressure of 3 Ttorr. Both the reactive PVD of TaN in pure nitrogen and Ta in pure argon resulted in similar rates of about 0.1 μm/min with maximum thickness in the centre of the coating spots. However, central parts of the spots can often contain large amounts of droplets from the melted spiral outlet. At higher RF powers the droplets from the melted sharp tip of the spiral can damage the coating and even melt the Si substrate. The heating of the spiral outlet was more intense in nitrogen than in argon. After 20 min also temperature of the sample table reached 500 °C in nitrogen plasma and up to 400 °C in argon. The sharp cut of the wire at the outlet of spiral can increase the local electric field and intensify eroding of the sample. Similar effect was confirmed by sharp ended stainless-steel medical needle with 1 mm outer diameter used as the hollow cathode.

5:20pm **IA1-MoA-12 Improvement of Surface Adhesion of Fluoropolymer Using Linear Ion Beam Source***,* ***Sunghoon Jung (hypess@kims.re.kr)****, J. Yang, E. Byeon, D. Kim, S. Lee, J. Park,* Korea Institute of Materials Science, Republic of Korea

Fluoropolymers, known for their excellent chemical and thermal resistances and low dielectric constants, play a pivotal role across diverse sectors. The inherent low surface energy of fluoropolymers, however, presents a notable challenge in terms of compatibility with other materials. Traditional methods to integrate fluoropolymers with different substances have largely relied on sodium-based chemical etching. These methods, while effective, often compromise the surface smoothness and are not environmentally sustainable.

In this study, we propose an innovative technique for the surface enhancement of fluoropolymers utilizing a linear ion beam source. By meticulously adjusting the ion beam process parameters, we have developed fluoropolymer bases with significantly improved hydrophilic characteristics. Additionally, this advanced technology has successfully increased the adhesive strength between fluoropolymer surfaces and the copper layers in flexible copper-clad laminates. The adoption of this novel surface modification method holds immense potential, especially in fabricating components for next-generation 6G mobile communication technologies, where strong and reliable adhesion is critically important.

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.

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2. Toboła D. et al., *Appl. Surf. Sci.*, 515 (2020) 145942
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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.

Surface Engineering - Applied Research and Industrial Applications
Room Town & Country C - Session IA2-2-TuA
Surface Modification of Components in Automotive, Aerospace and Manufacturing Applications II

Moderators: Vikram Bedekar, The Timken Company, USA, **Satish Dixit**, Plasma Technology Inc., USA

2:00pm **IA2-2-TuA-2 Impact of Novel Thermal Spray Material Solutions for Future Aerospace Applications and the Impact on Sustainability for the Environment and Business***, **Matthew Gold (matthew.gold@Liberty.Rolls-Royce.com)**,* Rolls-Royce North America **INVITED**

This presentation discusses the sustainability of materials and processes as applied to surface solutions in gas turbine engines.With the Aerospace industry under increasing pressure to improve the environmental performance of gas turbines, there is a growing need to reduce emissions and improve efficiency.This paper will outline the challenges associated with conventional materials and processes as well as the future materials that are being considered.

With an increase in turbine temperatures industry is moving towards more advanced materials systems for survivability.Over the last decade, industry has increased its use of rare earth oxides in thermal barrier coatings to help overcome the challenge of survivability in this harsh environment.This advance in materials comes with an impact on sustainability for the environment and business.

This presentation discusses these advanced materials for future applications and the challenges that will be encountered for sustainability.This will include raw materials, abundance, availability, and the need to understand the impact of process efficiency on their usage.

2:40pm **IA2-2-TuA-4 Evaluation of Thick Erosion-Resistant TiCrN Coating Deposited on Engine Impellers***, Q. Wang,* The University of British Columbia; Aurora Scientific Corp, Canada*; L. Hsu,* Aurora Scientific Corp, Canada*; **Da-Yung Wang (dayung.wang@ubc.ca)**,* The University of British Columbia, Canada; Aurora Scientific Corp, Canada; SurfTech Corp, Taiwan;, Canada

Metal-nitride hard coatings deposited through physical-vapor-deposition (PVD) techniques are increasingly being utilized in aircraft engines to protect compressor components against erosion caused by sand particles. Among these coatings, TiCrN, a ternary nitride coating with nano-layered configuration, exhibits promising results for application on turbine engine impellers to enhance erosion resistance. However, the deposition of TiCrN on impeller blades poses a unique challenge due to the sharp leading and trailing edges with curved airfoils, causing a shadowing effect during coating deposition. This can lead to non-uniform coating at sharp edges, resulting in spallation caused by high residual stress. To address this challenge, we employed various strategies, including a specially designed fixture providing two-axial rotation to the impeller, the incorporation of masking fingers to mitigate high coating deposition rates at sharp edges, modification of the ion cleaning process to enhance coating adhesion, and adjustments to chamber conditions such as increased working pressure using a mixture of N2 and Ar gases while reducing the substrate bias voltage to reduce coating residual stress.

The TiCrN coating, applied to a stainless-steel impeller and flat coupons by using cathodic arc deposition, underwent comprehensive characterization and testing. The coated impeller exhibited excellent surface coverage without spallation or cracking. The TiCrN-coated blades displayed consistent chemical compositions, and the surface roughness values (Ra) were maintained below 0.7 µm. The average hardness value of the coating was 2204 HV. The coating had excellent coating/substrate adhesion strength with critical loads higher than 40 N. Compared to the uncoated 1Cr11Ni2W2MoV substrate alloy, the TiCrN-coated blades demonstrated more than two times improvement in erosion resistance at 30°, 60° and 90° impingement angles. Furthermore, the TiCrN-coated samples exhibited no signs of corrosion damage after exposure to salt fog for 60 hours. In conclusion, the TiCrN coating applied to the stainless-steel substrate demonstrated exceptional performance in terms of erosion resistance, highlighting its potential for use in protecting turbine engineer impellers in aircraft engines.

4:00pm **IA2-2-TuA-8 Next Generation of Compositions & Coatings for Netzero & Sustainable Aviation***, **Tanvir Hussain (tanvir.hussain@nottingham.ac.uk)**,* University of Nottingham, UK **INVITED**

Thermal spray has proven to be a versatile coating deposition technique for many materials for wear, corrosion and thermal barrier applications; however, it is still challenging to spray oxygen-sensitive nano materials or carbides which sublimate in thermal spray.

Here we present a summary of various new approaches to deposit graphene nanoplatelet coatings and carbon nanotubes on their own from suspension and powder, as well as pre-mixed powders and composite suspension thermal spray. The new hardware modification and feedstock development allow direct incorporation of carbon-based nanomaterials into the thermal sprayed coatings that allow improvement in performance (for example, over two orders of magnitude in wear). Similarly, SiC is a cheap, abundant material for many engineering applications, including wear, but their lack of melting in a plasma or combustion flame in a desirable manner makes it very challenging to turn these into coatings. Here, we have developed a suspension and solution precursor process, a one-step route to produce composite coatings where SiC comes from suspension and the precursor salts (yttrium aluminium garnet here) transform into a protective matrix in the coating. This one-step process of suspension and solution precursor thermal spray has the potential to transform the materials portfolio of thermal sprayable materials.

Finally, axial injection suspension plasma sprayed coatings with columnar microstructures from ‘non-flammable’ organic solvent-based Yttria Stabilized Zirconia (YSZ) suspension will be introduced. The talk will cover the consequences of CMAS infiltration into these new coatings. The degradation of the coating mechanical properties due to CMAS ingression will be reported along with residual stresses using Raman spectroscopy. The common thread through all these examples will be reducing our CO2 footprint and improving component lifetime to achieve towards a netzero aviation.

4:40pm **IA2-2-TuA-10 Improved High Temperature Tribology for Aero-Engine Components by PVD Coatings***, **A.O. M. Eriksson (anders.o.eriksson@oerlikon.com)**,* Oerlikon Balzers, Oerlikon Surface Solution AG, Liechtenstein*; T. Middlemiss,* Oerlikon Balzers Coating UK Ltd., UK*; C. Jerg, E. Vaziri Beiraghdar, P. Kaller,* Oerlikon Balzers, Oerlikon Surface Solution AG, Liechtenstein*; T. Stelzig,* Oerlikon Balzers Coating Germany GmbH, Germany*; J. Ramm,* Oerlikon Balzers, Oerlikon Surface Solution AG, Liechtenstein

Aero engines operate under aggressive environments in which some of their components are exposed to temperatures well above 600°C. Under these conditions fretting and sliding wear is a major concern. For thermal management, dedicated materials like single crystal superalloys or Titanium aluminides are used in combination with protective coatings such as thermal barrier coatings and environmental barrier coatings. Besides thermal management, tribological behavior is important for engine components, such as shrouds, mechanical seals, rings, or joints, which are in contact with counterparts. Relative motion caused by vibrations is a common reason for fretting or sliding wear of the surfaces in contact. Pits and grooves on the contacting surfaces, as well as debris of removed material, may result in crack initiation and in failure of the component. Because the service temperatures are well above the application area for standard carbon-based tribological coatings, we have explored PVD coatings of oxides and nitrides. Coatings have been applied on superalloy specimens and tested in reciprocal sliding motion against superalloy counterparts at temperature of about 700 °C. The PVD coatings significantly reduced wear of the coated specimens, in contrast to extensive wear in the pairing of uncoated superalloy specimen with uncoated superalloy counterpart. Moreover, the evolution of the friction coefficient through the reciprocal sliding test was evaluated, where the coated specimen quickly stabilized at a constant value as opposed to the uncoated test conditions. The stable wear conditions are attributed to a tribologically transformed layer which was observed on the surface of the coatings, comprising components of the coating and superalloy material of the counterpart. The coatings can thus help to enhance lifetime and performance of tribologically loaded high-temperature components.

5:00pm **IA2-2-TuA-11 Development of Environmentally Friendly Solid Carburizing for Improving Fatigue Properties of AISI 4118 Steel***, Tomofumi Aoki (ao.tomofumi@keio.jp), D. Kasai,* Graduate School of Science and Technology, Keio University, Japan*; M. Hayama,* Keio University, Japan*; S. Takesue,* Kyoto Institute of Technology, Japan*; M. Tsukahara, Y. Misaka,* Neturen Co., Ltd., Japan*; J. Komotori,* Keio University, Japan

Gas carburizing is used extensively in the industry to improve the fatigue properties and the wear resistance of steel. However, the process is time-consuming and emits large amounts of gases, such as CO_2. Thus, we focused on atmospheric-controlled induction heating fine-particle peening (AIH-FPP) to resolve these challenges.

AIH-FPP combines induction heating and fine-particle peening. Shot media was projected onto a specimen heated with an IH coil. In AIH-FPP, when carbon is used as the projection media and steel is used as the base material, carbon can diffuse into the steel, and carburizing can be achieved rapidly. We named this process environmentally friendly solid carburizing, as it controls CO_2 emission and lessens the environmental burden. Accordingly, the aim of this study is to improve the fatigue properties of steel in a reduced time using this process.

The material used in this study was SCM 420H (AISI 4118 or equivalent) and the steel was machined into hourglass-shaped specimens. The air in the chamber was replaced with N_2 gas. The specimens were heated to 1273K for 30 s and then held at the temperature for 60 s. While heating and maintaining the temperature, steel particles coated with carbon were projected. The specimens were then cooled with N_2 gas. Afterward, these were requenched. We also prepared conventional gas carburized specimens.

An electron probe micro analyzer (EPMA) was employed to analyze the distribution of carbon concentration. The microhardness of each specimen was examined on their longitudinal section using a micro Vickers hardness tester. The fatigue test was conducted under axial loading with the stress ratio of -1 at room temperature, and test frequency of 10 Hz.

Environmentally friendly solid carburizing process diffused carbon up to 300 μm from the specimen surface and increased the carbon content on the specimen surface to approximately 0.5 mass%. No significant decrease in hardness was observed in the vicinity of the specimen surface. This result suggests that grain-boundary oxidation did not occur. This is because of the extremely low O_2 present in the treatment chamber, indicating that no CO was produced during the treatment. In addition, we consider that C_2H_2 is not produced during the treatment due to the displacement of the air with N_2 gas in the chamber. These results strongly suggest that the carbon diffused in this process by a mechanism different from the conventional gas carburization.

The fatigue life at the stress amplitude of 700 MPa was approximately 10 times longer than that of a conventional gas carburized specimen. This is because of the higher hardness on the specimen surface.

Surface Engineering - Applied Research and Industrial Applications
Room Palm 5-6 - Session IA3-WeM

Innovative Surface Engineering for Advanced Cutting and Forming Applications

Moderators: Stefan Bolz, CemeCon AG, Germany, **Denis Kurapov**, Oerlikon Surface Solutions AG Pfäffikon, Liechtenstein

8:00am **IA3-WeM-1 How to Design a Coating for Metal Sheet Deformation Starting from Cutting Tools***, **Alessandro Bertè (berte@lafer.eu)**, P. Colombi,* Lafer Spa, Italy **INVITED**

Metal sheet transformation processes still represent a fundamental sector of activity for the mechanical industry and for PVD coatings as well, as the latest have a primary role in reduction of production costs, thanks to the increase of the life of the mold and the reduction of friction during the molding phase.

Lafer has always invested in research for innovative surface treatments including coatings, aimed to improve the state of the art, in order to increase wear resistance of the moulds.

This research focuses on the mold throughout its entire life cycle, starting from its machining process and ending to its application on the field.

For this reason, the goal is twofold: namely to develop a coating for cutting tools used during mold construction while creating a high-performance coating for ferrous metal sheet deformation.

The starting point concerned the cutting tool: once the geometry and the material to be machined were defined (1.2379 steel hardened to 62 HRC), the influence of cutting edges preparation, coating and post-finishing techniques were investigated with the aim of minimizing tool wear.

Various coatings formulations on the market, specific for this application, were tested (AlTiN, AlCrN, AlTiSiN) and subsequently an AlTiSiN-based coating was developed using HiPIMS technology: the study allowed the improvement of the thickness uniformity, increasing the coating adhesion while optimizing its hardness and elastic modulus.

All the tested solutions were compared in terms of the wear of the cutting edges, finding that the HiPIMS AlTiSiN coating reached the best performances.

The second part of the project concerned the mold: a demanding geometry for ferritic metal sheet molding was identified and the cutting tools for the machining of the mold were prepared with the method defined above.

Field tests were carried out by comparing the uncoated mold against the current Lafer solution on the market (TiAlCN), based on the number of compliant produced parts.

Subsequently, a new TiCN-based coating deposited with arc technology was developed: the various tests led to a reduction of the friction coefficient and coating wear rate and increase of its fatigue resistance, measured through multiple impacts technique.

A molding comparison between the new and the actual solutions was carried out: the new coating led to a reduction in lubricant consumption and a significant increase in the number of produced parts.

Future developments will investigate the joint effect of a surface hardening process underneath the newly developed PVD coating and its performances with different types of metal sheet materials.

8:40am **IA3-WeM-3 Effect of Current Density on the Pulsed-DC Powder-Pack Boriding Process (PDCPB)***, I. Campos-Silva,* ***J.L. Rosales-Lopez (jrosales96@hotmail.com)****, M. Olivares-Luna, K. Chaparro-Pérez, E. Hernández-Ramírez,* Instituto Politécnico Nacional, Mexico*; A. Contreras-Hernández,* Tecnológico Nacional de México/Instituto Tecnológico de Tuxtepec, Mexico

In this study a novel method denominated pulsed-DC powder-pack boriding process (PDCPB) was used to develop FeB/Fe_2B layers on the surface of an AISI 316 L steel. The layers were obtained at 1123 - 1223 K, exposure times of 1800 – 7200 s for each temperature, employing current densities around of 230 and 460 $mA\cdot cm^{-2}$ with polarity inversion changes of the electric field of 10 s over the material's surface. A boriding agent composed by 70% B_4C, 20% SiC and 10% KBF_4 was used for the entire set of experimental conditions. The diffusion/electromigration-controlled growth of the FeB/Fe_2B layers on the AISI 316 L steel was validated by the "Mean Diffusion Coefficient" model. A positive effect of the current density on the boron activation energies of FeB and Fe_2B (~157 and ~165 $kJ\cdot mol^{-1}$ for the results obtained on 230 $mA\cdot cm^{-2}$, and ~150 and ~147 $kJ\cdot mol^{-1}$ at 460 $mA\cdot cm^{-2}$, respectively) were obtained; these results were lower (20% for the current intensity of 230 $mA\cdot cm^{-2}$ set and 26% for the current intensity of 460 $mA\cdot cm^{-2}$, respectively) than those reported for conventional powder-pack boriding process.

Furthermore, during the heating and electric field induction periods of the PDCPB, the temperature between the electrodes (T_E) and the electrical resistivity of the boriding agent (E_R) were sampled. In the same way, the induction of the electric field during PDCPB was possible due to KBF_4 percolation in the boriding agent and the transformation of part of the electron flux kinetic energy into heat (Joule effect), reducing drastically the E_R.

9:00am **IA3-WeM-4 Challenges Dealing with Industrial Coating Development and Tailor-Made Production***, **Klaus Pagh Almtoft (kpa@dti.dk)**, B. Christensen,* Danish Technological Institute, Denmark **INVITED**

The materials requirements of manufacturing industries encourage job coating companies to develop and adapt improved coatings or customize existing ones for emerging markets. The combination of coating research and development (R&D) for industrial applications, tailor-made coating solutions and job coating presents several practical challenges and opportunities.

One of the primary challenges in practice is translating R&D outcomes into scalable production processes. While the laboratory environment enables precise coating control and optimization, replicating these conditions on an industrial scale often proves difficult. Factors such as process variability, equipment limitations, the influence of complex geometries of real parts, and time and cost constraints can significantly impact the feasibility of implementing new coating technologies and processes. Different time scales between R&D and production may further complicate the process. In the pursuit of a solution, understanding every detail of the underlying science is not always feasible. A pragmatic approach can expedite the development process but requires a high expertise to avoid pitfalls.

Tailor-made solutions, while offering a high degree of customization, come with their own set of challenges. In daily PVD job coating operations, low volume production of certain parts demands flexibility and often non-standardized batches – frequently with mixed types of parts. This increases the need for skilled and highly experienced people to ensure consistent coating quality and performance. Additionally, coating processes developed for such applications need to be sufficiently robust to ensure the desired functionality, even when the geometrical setup of the coating system varies. In some cases, the use of simulation tools aids in optimizing batch setups to ensure sufficient coating homogeneity or thickness on the critical surfaces of components.

Despite these challenges, the potential benefits of customized coating solutions are substantial and serve many customers having a limited number of parts. By aligning R&D efforts more closely with production realities, it is often possible to accelerate the development of tailor-made PVD coatings and improve their production adoption.

This talk discusses some of the complexities encountered in bridging the gaps between R&D and production, focusing on applied customized sputter coating solutions and regular job coating. Practical examples of R&D using industrial scale DCMS and HiPIMS coatings for machine components and tools will be used as cases.

9:40am **IA3-WeM-6 a Comprehensive Study of HiPIMS Coated Tool and Microtool Performance: From Edge Preparation to Micro-Machining Tests***, **Pablo Díaz Rodríguez (pablo.diazr@nano4energy.eu)**, J. Santiago,* Nano4Energy, Spain*; A. García,* Nano4Energy, Colombia*; I. Fernández, A. Wennberg,* Nano4Energy, Spain*; P. Collignon,* PD2i, France*; Á. Guzmán, D. Sanmartín, J. Molina-Aldeguia,* Universidad Politécnica de Madrid, Spain*; M. Monclus,* IMDEA Materiales, Spain

The high standards and requirements demanded in high-speed machining (HSM) applications – some examples are the precise manufacturing of IC Molds or biomedical devices - comprise a delicate control of the tool preparation as well as coatings design and finish, especially in the case of microtools with a diameter below the millimeter.

The work developed covers not only the coating step, but also the preparation of the tool, focusing on:

a) Microtool cutting edge preparation.

b) HiPIMS deposition, as the use of this technology allows the preparation of hard coatings with high smoothness, low density of defects, and good homogeneous coverage of 3D intricate parts (thus able to match the low tolerances required for micromachining) makes this technology ideal for these applications. The tested coatings are based on Si- and B- containing AlTiN and were deposited in different sets of tools, according to their specific requirements, attaining hardness values of 35 GPa and good adhesion. Moreover, oxidation studies were performed to determine the stability of these coatings, analysing, and comparing the results in terms of SEM, TEM, and XRD, observing a greater oxidation resistance for the Al containing coatings.

c) Machining tests, which, in addition to mechanical properties analysis, provide information regarding the performance of the coatings under operation conditions. The materials selected for machining are Hardened Steel (HRC60) and Ti6AlV4 alloy, and the finishing of the machined parts, as well as the wear suffered by the tool is analyzed.

11:00am **IA3-WeM-10 Effect of Phase Separation in the Anticorrosion Performance of AlCrFeNi High-Entropy Alloy**, ***Chih-Chen Lee (janislee0123.en12@nycu.edu.tw)****, I. Tasi,* National Yang-Ming Chiao Tung University, Taiwan*; H. Chen,* Michigan State University, Taiwan*; C. Chen,* National United University, Taiwan*; S. Chen,* National Yang-Ming Chiao Tung University, Taiwan

Due to reaching net-zero emissions, offshore wind power is one of the methods to get clean energy. To improve the anti-corrosion performance of wind turbine towers, researchers are always seeking the candidates to enhance or replace the stainless steel 316 base material. In our study, we found that AlCrFeNi HEA exhibited a better anti-corrosion performance than SS 316 in both the salt spray and acid immersion test. Especially, its corrosion resistance could be significantly improved by controlling the phase ratio. Gas-atomized HEAs can retain the ideal high-entropy state owing to the sluggish effect and rapid cooling process. The as-atomized AlCrFeNi powders presented a superior resistance to acid solutions, but weakened after experiencing high temperature environment. Through careful investigations, it was found that Al and Ni elements have the lowest mixing enthalpy, promoting the preferential formation of AlNi phase from the uniformly distributed matrix. As a result, ordered AlNi and FeCr phases are formed within the BCC structure. It has a chemical composition closely matching the designated component ratios, composed of a BCC/B2 phase composed of AlNi and a BCC/A2 phase composed of FeCr. From the acid immersion test, we found that the rich AlNi phases were preferentially corroded, decreasing corrosion resistance. Furthermore, argon gas was commonly used to atomize the melt and has a lower specific heat capacity, which allows sufficient time for melted droplets to form spherical shapes with better flowability due to cohesive forces. The AlCrFeNi HEA powder produced by gas atomization exhibits a good anti-corrosion performance because it maintains the initial high randomness phase and prevents the segregation of elements. It also shows a spherical shape and excellent flowability, making it suitable for coating applications in harsh environment.

Keywords: High-entropy alloys, Gas atomization, AlCrFeNi, Annealing, Phase transition, anti-corrosion performance

11:20am **IA3-WeM-11 Surface Conditioning and New Applications Using Advanced Plasma Etching Technology**, ***Dominic Stangier (dominic.stangier@oerlikon.com)***, Oerlikon Balzers Coating Germany GmbH, Germany

Plasma etching plays an essential role for the vacuum-based cleaning of tools and components to remove native oxide films and small contaminations. This inherent treatment of the substrate material prior to every PVD process directly influences the adhesion and consequently the overall performance of the substrate coating compound. Therefore, different processes such as glow discharges and metal ion etching methods are commonly conducted, which are however on the one side strongly limited in their etching rate as well as performance and on the other side lead to macro defects on the surface of the substrate significantly reducing the performance of the coated tools. To overcome these challenges an improved etching process, which combines the high plasma density of cathodic arc evaporation with a noble gas-based glow discharge called advanced Arc Enhanced Glow Discharge (AEGD) is used. In this context, the unique possibility to independently control the bias potential and freely modulate the pulse pattern with a simultaneous scalable plasma density for the etching process open up a broad field of new pre-treatment options for PVD coated tools.

Fundamental investigations for the limits and correlations of the aforementioned etching parameters on the surface condition, near subsurface region and the coating adhesion of nitrides are conducted using cemented carbide substrates. The etching rate is directly linked to the applied bias level as well as to the current used on the cathode and anode. As a result of the high etching rates, a new approach for a targeted cutting edge preparation for micro tools is presented, showing the possibility of generating asymmetric cutting edge shapes (form factors up to $K = 2.7$). The performance is evaluated in milling tests, proving a reduction of the process forces for milling HSS (62 HRC). Furthermore, for tool steels an adjusted composition of the plasma allows the nitriding of the surface near region, which also leads to improved performance of coating systems for dies and molds. Thus, the presented investigations prove the extended possibilities and application fields offered by the advanced AEGD technology.

11:40am **IA3-WeM-12 Advances in Microhard Machining: From Etching-based Asymmetrical Cutting Edge Preparation to Cutting Performance of TiAlN-based Thin Films**, ***Nelson Filipe Lopes Dias (filipe.dias@tu-dortmund.de)****, A. Meijer, C. Jäckel, D. Biermann, W. Tillmann,* TU Dortmund University, Germany

Micromilling of hardened and tempered tool steels offers significant potential for die and mold manufacturing due to the high precision in dimension and shape of filigree geometries combined with high surface integrity. Nevertheless, the high hardness of these steels impose considerable thermo-mechanical loads on the cutting edge of micromilling tools. Hence, it becomes essential to employ an adapted combination of cutting edge preparation alongside the application of wear-protective PVD thin films with improved properties. This integrated approach is crucial for enhancing cutting performance and prolonging the service life of tools when machining hard materials. Arc-enhanced glow discharge (AEGD) ion etching emerges as a promising pretreatment, serving not only to enhance thin film adhesion, but also to produce asymmetrical cutting edge geometries in small scale for micromilling tools of ultrafine-grained WC-Co cemented carbide. Already 15 min of AEGD ion etching yields in considerable cutting edge rounding and the formation of asymmetrical shapes with form-factors $K \geq 2$. The obtained cutting edge geometries promote favorable cutting behavior in terms of process forces and wear development in machining tests of a hardened and tempered powder metallurgical high-speed steel (AISI M3:2). In addition to the AEGD ion etching pretreatment, the effectiveness of micromilling tools substantially relies on the application of a protective PVD thin film with enhanced tribo-mechanical properties. Both the selection of the TiAlN-based thin film system and the employed sputtering technology play pivotal roles in determining the cutting performance of the coated tools. In comparison to the traditional TiAlN, quaternary TiAlSiN and TiAlTaN thin films exhibit superior wear resistance for micromilling tool steels with high hardness and carbide content. The use of high power impulse magnetron sputtering (HiPIMS) further enhances cutting performance and wear resistance, particularly for TiAlSiN. To overcome the low deposition rates of pure HiPIMS processes, a hybrid approach combining direct current magnetron sputtering (dcMS) with HiPIMS is a viable alternative, taking advantage of the benefits of both processes. This hybrid dcMS/HiPIMS technique also proves beneficial in producing TiAlN-based thin films that exhibit improved cutting performance and wear resistance compared to pure dcMS thin films. These findings emphasize the importance of a coordinated approach involving ion etching and PVD deposition for effectively reducing wear and process forces in micromilling difficult-to-machine materials.

Surface Engineering - Applied Research and Industrial Applications
Room Golden State Ballroom - Session IA-ThP

Surface Engineering - Applied Research and Industrial Applications (Symposium IA) Poster Session

IA-ThP-1 Application and Practice of Surface Aluminization Treatment in Zinc Pot Equipment of Hot Dip Galvanizing Production Line*, **Lu Wang (4986208@qq.com)***, BAOSTEEL, China

hot dip galvanizing is a process of coating the surface of a steel strip with a zinc layer to prevent corrosion. This process is widely used in industries such as automobiles, home appliances, and construction. During the hot dip galvanizing process, various components on the galvanizing line are immersed in the high-temperature molten zinc liquid in the zinc pot, which has a certain degree of corrosiveness and can cause corrosion to components such as sink rolls, stabilizing rolls, zinc pumps, and snout. In the continuous hot dip galvanizing process of strip steel, due to the corrosiveness of the high-temperature molten zinc liquid in the zinc pot, the service life of some components in the galvanizing equipment is concise, with an average service life of only 12-15 days. This seriously restricts the production efficiency of continuous hot dip galvanizing, increases economic costs, and also affects product quality. Parts in direct contact with high-temperature molten zinc on the galvanizing line are required to resist zinc corrosion and thermal shock.

The use of thermal spraying technology for surface coating treatment of components in zinc pots can have a certain anti-corrosion effect, but it cannot be widely used due to its high cost. This article introduces an aluminizing technology that involves placing components in a molten aluminum pot for hot dip aluminum pot, and then diffusing at a temperature of 800-950 ℃ to transform all the aluminum plating layers on the hot-dip aluminum surface into aluminum iron compound layers, forming a diffusion type aluminizing layer. This thin film can effectively prevent the corrosion of zinc solution on components and also inhibit the formation of zinc dross on the surface of components.

Through experiments, we found that after muffle furnace annealing, a uniform and dense Al2O3 film is formed on the surface of aluminized stainless steel. Aluminum oxide has unique properties that metal and organic polymer materials do not have. Aluminum oxide films have excellent wear resistance, corrosion resistance, heat resistance, high-temperature oxidation resistance, insulation, and other properties. The Al2O3 film isolates the steel substrate from the zinc liquid, preventing mutual diffusion and reaction between Fe and Zn atoms. The Al2O3 film serves as an isolation layer, which can prevent corrosion of the steel substrate by zinc liquid. The cost of this surface aluminizing treatment is much lower than that of thermal spraying, which not only prolongs the service life of the components but also significantly reduces maintenance costs.

IA-ThP-5 e-Poster Presentation: Bismuth Thin Film Electrodes*, B. Frontana-Uribe, V. Ugalde-Saldivar, A. Hernandez-Gordillo, A. Vazquez,* Universidad Nacional Autónoma de Mexico*; **Sandra E. Rodil (srodil@unam.mx)***, Universidad Nacional Autonoma de Mexico

Bismuth film electrodes (BiFE) for trace metal detection using electroanalytical techniques have been researched since 2000, after the demonstration that the BiFE could substitute mercury drop or mercury film electrodes, leading to a safer and eco-friendly solution. However, after more than 20 years of research, the BiFEs are not yet available for commercial use. In this work, bismuth-based thin films produced by magnetron sputtering have been tested for detecting trace metals and organic molecules of interest. Moreover, the stability of the Bismuth-based electrodes in different non-aqueous solutions has been studied, aiming to use the electrodes for the electrosynthesis of organic molecules.

Pure bismuth, bismuth-tin, and bismuth-indium films were deposited on both smooth and rough glass substrates. These were used as the working electrodes in a three-electrode electrochemical cell, where different electroanalytical techniques were used to detect metal ions or organic molecules of interest, such as acetaldehyde. The same electrodes were also tested for the electrosynthesis of organic molecules, which constitutes a sustainable method to produce high-value chemicals without using catalysts.

The results are summarized to present the potential use of bismuth-based electrodes produced by a physical vapor deposition technique for detecting cadmium, zinc, acetaldehyde, and insulin. The Bi-In electrode was tested to drive the cathodic reduction of benzophenone using Cyrene™/EtOH (1 : 1) as a green solvent mixture. Interestingly, the Bi–In electrode yielded a 56% of the pinacolic compound. Such reaction can be used to prepare alcohols and diols from the electrochemical reduction of carbonyl compounds, such as aldehydes and ketones.

IA-ThP-6 Fabrication of TiO_2 Nanotube/SiNW Arrays Structure at Different Synthesis Parameters for Solar Cell Application*, **Ai-Huei Chiou (ahchiou@gs.nfu.edu.tw)**, Z. Lin,* National Formosa University, Taiwan

Titanium dioxide is renowned for its non-toxicity, high chemical stability, and excellent photocatalytic activity, making it widely applicable in areas such as photocatalysis, photodegradation, and solar energy-related applications. Various methods, including hydrothermal synthesis, sol-gel techniques, and anodization, can be employed to obtain titanium dioxide nanostructures. Among these methods, anodization is favored by many researchers for its simplicity, cost-effectiveness, and ease of observation.

This study utilizes the anodization method to prepare a novel hybrid silicon nanowire array structure and explores its feasibility for application in solar cells. The research primarily focuses on the preparation of titanium dioxide nanotube structures, comparing the results of nanoscale structures with non-nanoscale structures in solar energy measurements.

Currently, most anodization methods used for preparing titanium dioxide nanotubes utilize platinum metal as the cathode, despite its better stability, it is expensive. The anode is typically made of pure titanium foil or sheet. In this study, a novel structure is proposed, involving the deposition of a seed layer on the anode silicon nanowire array, and using a pure titanium plate as the cathode for anodization. The study investigates structural changes under different experimental parameters.

The research employs a trial-and-error approach to sequentially adjust parameters such as electrolyte water content, current, voltage, and film thickness to confirm the conditions for subsequent anodization. A magnetron sputtering machine is used to deposit titanium on the silicon nanowire array, and finally, anodization is employed to prepare a divergent structure of titanium dioxide nanotubes.

SEM observations indicate that with appropriate water content, current, voltage, and film thickness, a complete pore morphology can be obtained. Raman analysis reveals TiO2 lattice peaks under different growth times, confirming the prepared TiO2 has a rutile structure. Additionally, UV-Vis analysis shows that when the substrate is non-nanoscale, the reflectance is approximately 80%, but when the substrate is a silicon nanowire, the reflectance decreases with increasing TiO2 thickness. In terms of electrical properties and solar energy analysis, the TiO2 nanotube/Si structure demonstrates a conductivity of 8.856×10^{-7} S/cm and a photovoltaic conversion efficiency of 2.31×10^{-3}, while the TiO_2 nanotube/SiNW Arrays Structure exhibits a similar conductivity of 8.856×1010^{-7} S/cm and a higher photovoltaic conversion efficiency of 5.46×10^{-3}.

IA-ThP-7 Process-Awared Compact Modeling to Obtain Consistent Performance for Various Gate-All-Around Structures Due to Vertical Oxidation and Etching Process of 3D Charge Trapping Flash Memory*, **Sunghwan Cho (joboss9999@gmail.com)***, Samsung Electronics and Department of Semiconductor and Display Engineering, Sungkyunkwan University, Republic of Korea

To address the scaling limitations of conventional planar flash memory, gate-all-around (GAA) charge trapping flash (CTF) memory has emerged as the most promising alternative, offering significantly larger storage capacity and reduced disturbance. However, as more layers are stacked vertically and feature sizes are decreased, it becomes increasingly challenging to manage vertical processes like etching or oxidation, leading to variations in geometry such as hole radius or tunneling oxide thickness, respectively. Furthermore, unexpected process variations along the word lines (WLs) pose challenges for circuit designers in optimizing conditions for consistent performance, primarily due to the absence of a framework based on circuit simulation. Therefore, in this study, we introduce a compact modeling approach aimed at delivering optimized solutions for achieving uniform performance after program and erase operations along the WLs, thereby reducing the effects of process variations inherent in vertical GAA structures within 3D CTF memory devices such as hole etching and vertical oxidation. The program and erase performance exhibit unexpected differences due to variations in the etching process and vertical oxidation within gate-all-around structures, resulting in variations in hole radius and tunneling oxide thickness along the WLs, as shown in Fig. 1 and 2. We presented diverse variations in hole radius and tunneling oxide thickness across three representative GAA structures (trapezoid, entasis and iterated trapezoid in Fig. 1). Then, utilizing the proposed workflow outlined in this paper, we extracted optimized WL gate biases to ensure a uniform electric field along the WLs, as shown in Fig. 3. Finally, we presented the variation in program speed across different locations within a string and demonstrated consequent uniform performance using SPICE simulation, as shown in Fig. 4. As a valuable framework, the proposed compact modeling enables circuit designers to optimize design schemes for consistent behavior in 3D CTF memory devices.

IA-ThP-8 Disruption of Cell Wall Using Non-Thermal Plasma for Recovery of Intracellular Lipid to Be Used as Bio Lubricant*, JOSÉ GERALDO PRADELLA (jpradella51@gmail.com),* Universidade do Vale do Paraiba, Brazil

Rhodosporidium toruloides stands out as a highly promising oleaginous yeast, renowned for its capacity to amass substantial quantities of intracellular neutral lipids. These lipid reservoirs hold significant commercial value across diverse industries such as biofuels, food production, chemicals, pharmaceuticals, and bio lubricants. Leveraging non-thermal plasma for yeast cell wall disruption presents a distinct advantage in preserving the integrity of these sought-after intracellular components. The cultivation process of this oleaginous yeast involves two distinct stages, each utilizing specific culture media. The inoculum phase employs Saboraud medium, while the production phase utilizes a defined medium with a C/N optimized ratio. Following the fermentation stage, the harvested material undergoes centrifugation, with the discarded supernatant paving the way for subsequent non-thermal plasma treatment. This plasma process, executed in triplicate with five samples, incorporates variations in exposure time—ranging from 5 to 30 minutes—while keeping other parameters constant. Characterization of the samples, both before and after plasma treatment, involves comprehensive analysis using scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), and tribometer assessments. Notably, tribometer evaluations employ the treated samples as a biolubricant interface between a 316L plaque and sphere. The results display a progressive disruption of the yeast cell wall upon exposure to non-thermal plasma, with noticeable effects emerging as early as 5 minutes and culminating in complete destruction within the 30-minute timeframe. The FTIR was used to analyses the bio lubricant structure before and after friction tests. This study underscores the immense potential of non-thermal plasma as a groundbreaking technique for efficiently extracting lipids through cell wall disruption, thereby contributing to the advancement of sustainable bioprocessing and bio lubricant production.

Surface Engineering - Applied Research and Industrial Applications
Room Town & Country D - Session IA2-3-FrM
Surface Modification of Components in Automotive, Aerospace and Manufacturing Applications III
Moderator: Ta-Chin Wei, Yuan Christian University, Taiwan

8:00am **IA2-3-FrM-1 Study of Piezo-photocatalytic Performance of p-CoS-n-NaNbO₃ Junction Composite***, Man-Yu Hsiao (n56121288@gs.ncku.edu.tw), T. Nguyen, K. Chang,* National Cheng Kung University (NCKU), Taiwan

Piezoelectric materials have been applied to the application of photocatalysis, photoelectrochemical cells, and pressure sensors. Furthermore, their heterojunction composites can enhance the photocarrier separation through a built-in electric field, and induced piezoelectric potentials can also minimize photocarrier recombination, improving photocatalytic efficiencies. In this talk, detailed studies on piezoelectric $NaNbO_3$, CoS-$NaNbO_3$ composites, and their associated piezo-related applications will be discussed. Various morphologies of $NaNbO_3$ were tuned via facile hydrothermal methods by the adjustments of heating times and temperatures, solution concentrations, and precursor types (Fig. 1-3, Supplement). Crystal phases of the samples were determined using XRD. Morphology and microstructures of $NaNbO_3$ and their composites were examined by SEM and TEM. Optical properties of the samples were investigated using UV–Vis spectroscopy. The sample’s conductivity types were determined through Mott-Schottky measurement. Piezoelectric properties were directly measured via piezoresponse force microscopy (PFM). Furthermore, piezo-photocatalytic applications of $NaNbO_3$ and CoS-$NaNbO_3$composites were also explored, and the performance was elucidated using a constructed energy band diagram. Our results reveal that the photocatalytic effectiveness of the CoS-$NaNbO_3$ composite is attributable to the robust formation of p–n junctions, piezoelectric potentials, substantial amounts of active surface areas, and band positions.

8:20am **IA2-3-FrM-2 Enhanced Metal Surface Finishing with EPPo: Innovative Strategies for Ti 6Al-4V Alloys***, Nicolas Laugel (nicolas.laugel@manchester.ac.uk), A. Matthews, A. Yerokhin,* The University of Manchester, UK

Electrolytic plasma polishing (EPPo) emerges as a promising technique for the precision removal and refinement of metals' surface layers. Its applications are experiencing growing interest, particularly in improving the finishing of lightweight alloys produced through additive manufacturing (AM). While the AM of metals holds immense potential to revolutionize global production, it faces a persistent challenge of achieving satisfactory final surface quality. To tackle this challenge, effective post-treatment methods are crucial, and EPPo emerges as an ideal solution.

The EPPo process involves placing the workpiece as the anode in an electrolytic cell and applying DC voltages in the hundreds of volts. The resultant energy release at the interface induces various physical and chemical reactions, ultimately influencing the processes governing the working surface’s dissolution and allowing more precise control. EPPo employs electrolyte compositions that are safer for the workplace and the environment compared to traditional electropolishing methods, albeit with a slower material removal rate. Nevertheless, it preserves the advantages of contactless and geometry-independent polishing, aligning ideally with the advantages of additive manufacturing.

EPPo holds particular significance in its application on titanium and its alloys, a class of materials highly valued across various industries for their exceptional strength-to-weight ratio, corrosion resistance, and biocompatibility. They also present a valuable testing ground for EPPo due to their challenging electrodissolution characteristics, particularly the interference caused by the formation of insoluble oxides that create passivation layers, hindering the EPPo process itself in a positive feedback loop. This investigation specifically focuses on the impact of electrolyte on the surfaces generated through the EPPo of Ti 6Al4V alloy.

As EPPo has been transitioning from laboratory to industrial scales in recent years, electrolyte replacement has emerged as a significant and unexpected obstacle to cost-effective implementation. Notably, considerable downtime arises with the associated waste management tasks and inertia in temperature change of larger volumes. This study explores novel, environmentally friendly, non-aqueous electrolytes with deep eutectic solvents and their unique advantages in the application of EPPo to valve metals such as titanium and aluminium. Furthermore, it examines the ageing process in conventional fluoride-based aqueous electrolytes, providing insights from both chemical and surface science perspectives, along with proposed mitigations.

8:40am **IA2-3-FrM-3 Optimization of Plasma Electrolytic Polishing for 304 Stainless Steel Using Taguchi Method***, Chun-Wei Chang (yiwenz988@gmail.com), N. Zheng, C. Tseng,* Ming Chi University of Technology, Taiwan, Republic of China

Plasma electrolytic polishing (PEP) is an advanced and efficient surface treatment technique widely employed across various industries. These particles induce chemical reactions on the material surface, thereby achieving fine polishing and enhancement of surface properties. In this study, we focused on utilizing a low concentration of ammonium sulfate ($(NH_4)_2SO_4$) aqueous solution to perform plasma electrolytic polishing on 304 stainless steel surfaces. The Taguchi experimental method was employed to assess the effects of different PEP parameters such as process time, voltage, electrolyte concentration, and temperature on the surface roughness,wettability and corrosion resistance of the PEP-treated stainless steels. Optical microscopy (OM) and scanning electron microscopy (SEM) were utilized to capture the changes in surface defects on 304 stainless steels after PEP processes, while a white light interferometer was used to evaluate surface roughness and flatness. Water contact angle measurements were conducted to assess the hydrophilicity/hydrophobicity of the PEP-treated 304 stainless steels. Additionally, the corrosion resistance properties were evaluated by using potentiodynamic polarization curve measurements in 3.5 wt% NaCl solutions. The experimental results performed by Taguchi analysis reveal that the electrolyte concentration is the most significant parameter affecting the effectiveness of plasma electrolytic polishing for 304 stainless steels, and followed by process time, voltage, and temperature. The OM and SEM images indicate that the removal of surface defects is increasing by increasing the process time, maximum voltage, and electrolyte concentration. The experimental data measured by the white light interferometer reveal that the lowest surface roughness of 0.061 μm and optimal surface flatness are achieved at PEP parameters of 6 minutes, 320 volts, and 5 wt% ammonium sulfate concentration. However, the wettability of PEP-treated 304 stainless steels obtained by water contact angle measurements indicates that an enhancement in hydrophobicity for PEP-treated surfaces, with the contact angle increasing from 73.8° before PEP to a maximum of 96.6° after PEP in 5 wt% ammonium sulfate solution. Furthermore, the pitting resistance for PEP-treated 304 stainless steels evaluated by potentiodynamic polarization curves measured in 3.5 wt% NaCl solution demonstrates that the highest pitting nucleation potential (E_{np}) and the largest passive region, indicating superior corrosion resistance properties, achieve on PEP-treated surface after PEP in 5 wt% ammonium sulfate solution.

9:00am **IA2-3-FrM-4 Structure Design and Degradation Mechanism of Amorphous Carbon Coatings on Metallic Bipolar Plates***, Hao Li (lh@nimte.ac.cn), P. Guo, A. Wang,* Ningbo Institute of Materials Technology and Engineering, Chinese Academy of Sciences, China

Proton exchange membrane fuel cell (PEMFC) is important on the development of hydrogen energy and fuel cell technology. However, in the acidic working environment of the PEMFC, the metallic bipolar plates (BPs), as a core component in PEMFC, will face to the problems of dissolution and corrosion, which directly determines the output power and service life of the PEMFC. Therefore, improving the conductivity and corrosion resistance of the metallic BPs is one of the key technologies that need to be tackled in the PEMFC field, affecting the competitiveness of the hydrogen energy and fuel cell industry.

The amorphous carbon (a-C) coating can endow metallic BPs with excellent corrosion resistance and conductivity. Besides, the scale cost advantage of a-C coating is significant. It can be prepared by physical vapor deposition technology, and has great application potential in metallic BPs of PEMFC. However, the preparation of a-C coating with excellent performance and the realization of stable low interface contact resistance (ICR) are still great challenges in this field. In addition, the degradation mechanism of a-C/metallic BPs after long-term operation is not clear, which seriously limits the development of corrosion-resistant and conductive a-C materials and technologies.

In view of the above problems, this paper fabricated a series of a-C coatings for the surface modification of metallic BPs under PEMFC conditions, and explored the relationship between the microstructure and bonding composition of coatings and corrosion resistance and conductivity, revealed the failure mechanism of the coatings modified metallic BPs, prepared a-C coating with high conductivity and corrosion resistance, and put forward a new idea of interface optimization of the coating modified metallic BPs.

9:20am **IA2-3-FrM-5 Automated Laser Cleaning/Ablation as a Novel Tool in Aerospace Manufacturing**, ***Dmitri Novikov (dnovikov@ipgphotonics.com)***, IPG Photonics, USA

This presentation will explore the progress in laser technology that has made them the tool of choice for mass manufacturing, with a focus on laser cleaning/ablation. Laser technology has revolutionized the way we manufacture, maintain, repair, and overhaul aerospace components. Thorough cleaning is a critical step in these processes, whether before coating, surface polishing or roughening, or any joining operations like welding or brazing. Currently, three main technologies are used for coating stripping and surface preparation for coating: abrasive grit blasting, abrasive water jetting, and chemical cleaning/stripping. However, these technologies negatively impact the environment and health and are slow and expensive. This presentation aims to introduce a laser cleaning solution that can replace these legacy technologies. Although laser cleaning/ablation is known in the industry, its use is limited due to the limited access to correct laser sources and concerns of part damage by laser heat. The presentation will showcase successful laser cleaning applications for different cleaning/ablation tasks, resulting in improved productivity, repeatability, direct cost savings, and part performance improvements in quality. Laser technology has proven to be a game-changer in the manufacturing industry.

Author Index

Bold page numbers indicate presenter