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

Moderators: Dr. Satish Dixit, Plasma Technology Inc., USA, **Masaki Okude**, Mitsubishi Materials Corporation, Japan, **Dr. Jan-Ole Achenbach**, KCS Europe GmbH, Germany

1:40pm **IA2-1-MoA-1 Laser Surface Remelting Induced Reaction Sintering of Nickel and Titanium Powders**, ***Milton Lima [miltonsflima@gmail.com]***, Institute for Advanced Studies, Brazil; *Alana Brito,* Technological Institute of Aeronautics, Brazil; *Felipe Costa,* BRENG Co., Brazil; *Rafael Siqueira,* Technological Institute of Aeronautics, Brazil; *Sheila Carvalho,* Federal University of Espirito Santo, Brazil

There is currently an interest in the synthesis and applications of mnemonic structure materials for various applications, such as biomedical, aerospace, automotive, and robotics. Shape memory alloys (SMAs) are metallic materials that can return to their initial state after being subjected to deformation as a result of increased temperature, increased pressure, or other stress conditions. These materials have been used in thermoelastic actuators in space applications, such as antenna supports and solar panel deployments, and can impact the manufacture of polymorphic aircraft engines and fuselages. SMAs, such as Nitinol (equimolar alloy of Ni and Ti), are difficult to fabricate, and the powder metallurgy route, whether classical or using laser powder bed fusion (L-PBF), has been constantly improved to meet new application niches. This study proposes reactive sintering of samples with equiatomic compositions of Ni and Ti to form Nitinol using laser surface remelting. Elementary powders were inserted into a high-energy ball mill to fabricate mechanically alloyed Ni-Ti powders, which were subsequently pressed in the form of discs (20 mm diameter, 5 mm thickness). An experimental arrangement with a vacuum chamber and fiber laser beam manipulation was developed to induce sufficient heat for the reaction of elementary powders. Spiral scanning of the fiber laser beam produced surface remelting that ignited the pressed powder mixture. Macro- and microstructural analyses, the crystalline structure, and the composition of the sample surface remelted with a beam power ranging from 10 to 46 W were performed. In this power range, the sintering time was varied between 55 and 295 s when the laser power was varied from 46 W to 10 W. Although the presence of intermetallic phases was approximately the same, the microstructure in the laser-surface-remelted region was more homogeneous than that in the sintered volume. At the end of sintering, tablets were obtained with an apparent density of 61%–67% and a large number of intermetallic phases, such as $NiTi_2$, Ni_3Ti, and Ni_4Ti_3, together with unreacted elemental powders (Ni and Ti). The samples prepared in air also presented these phases in addition to TiO_2 and NiTi. The air-processed samples presented an equimolar Nitinol phase, as observed by X-ray diffractometry. According to mass spectrometry analyses of secondary ions, the presence of air oxidized the surface of the grains, which reacted at shorter distances and generated the Nitinol phase.

2:00pm **IA2-1-MoA-2 A Comparative Study on the Formation of Micro-Arc Oxidation Coatings on AZ31 and AC84 Magnesium Alloys**, ***Chi-Hua Chiu [qiuqihua90@gmail.com]***, *Shih-Yen Huang, Yueh-Lien Lee, Yu-Ren Chu,* National Taiwan University, Taiwan

Magnesium-aluminum-calcium (Mg−Al−Ca) alloys have attracted significant attention due to their excellent strength-to-weight ratio, good castability, and potential for flame retardancy, owing to the presence of calcium and aluminum. However, the applications of these alloys are limited by their poor corrosion resistance. Micro-arc oxidation (MAO) is one of the most common techniques for corrosion protection of magnesium alloys; however, the formation mechanism of MAO coatings is extremely complex and influenced by numerous process parameters, including the substrate effect. In this study, the mechanism of MAO coating formation on the AZ31 and dual-phase AC84 (Mg−8Al−4Ca) alloys was comparatively examined. The preliminary results reveal that, during MAO treatment at a constant anodizing voltage of 150V, unreacted Al_2Ca secondary phases were observed in the micro-arc oxidation coatings of AC84 magnesium alloys, causing non-uniform surface structures and thicknesses, which led to poor corrosion resistance compared to the MAO coating formed on AZ31.Conversely, AC84 exhibited better corrosion resistance than AZ31 when the voltage was increased to 250V. Further increasing the voltage to 300V resulted in the involvement of secondary phases in the reaction, leading to more uniform microstructures and chemical compositions of the coatings on both alloys. These findings suggest that the anodizing voltage plays a crucial role in the reaction behavior of secondary phases and the properties of the MAO coatings.

2:20pm **IA2-1-MoA-3 Ultra-High Vacuum Test System for Quantitative Determination of Hydrogen Permeability of Various Ceramic Coatings on Stainless Steel**, ***Ewa Rennebro [ewa.ronnebro@pnnl.gov]***, Pacific Northwest National Laboratory, USA **INVITED**

We will discuss a recently built state-of-the-art ultra-high-vacuum (UHV) test system with high accuracy and precision to quantify hydrogen uptake, solubility and diffusion in various materials. The design was developed by Pacific Northwest National Laboratory (PNNL) in collaboration with Vacuum Technology Inc (VTI). This automated UHV system can be used for several studies of hydrogen-metal interactions including absorption/desorption kinetics, thermodynamics, isotherms, plateau pressures, isotope studies, gaseous impurity identification and permeation rate. We will present recent permeation rate data of ceramics coatings to reduce permeation through stainless steel.

3:00pm **IA2-1-MoA-5 HIPIMS – Fascinating Technology to Make Next Steps in Tool, Decorative and Functional Applications**, ***Philipp Immich [pimmich@hauzer.nl]***, *Ivan Kolev, Andreas Fuchs, Daniel Barnholt, Julia Janowitz, Louis Tegelaers, Huub Vercoulen, Chinmay Trivedi, Geert-Jan Fransen,* IHI Hauzer Techno Coating B.V., Netherlands; *Holger Hoche, Thomas Ulrich,* TU Darmstadt, Germany; *Peter Polcik,* Plansee Composite Materials GmbH, Australia

The PVD (Physical Vapor Deposition) market is rapidly expanding into new application fields. To achieve these new applications, various PVD coating techniques are employed, with HIPIMS (High Power Impulse Magnetron Sputtering) being one of the most fascinating since its discovery. Over the past 25 years, numerous advancements have been made in latest Generation 3 - HIPIMS power supply technology, including modifications in bipolar mode, pulse shape, pulse length, pulse trains, and higher frequencies. Synchronization of cathodes and HIPIMS-based bias has also led to innovative PVD coating solutions.

Beyond the well-known performance improvements in HIPIMS-coated cutting tools, HIPIMS has demonstrated its potential for various other applications. We will showcase the ability to create different colors using HIPIMS technology and highlight its advantages for decorative applications on 3D products. For components, HIPIMS is an excellent tool for enhancing the wear and corrosion resistance of existing material systems. We will also present the latest coating development for cutting tools. Our presentation will illustrate how combining HIPIMS with new material systems can further expand and enhance potential application areas.

Decorative, tool and tribological markets are driven by production costs, making coating volume and size crucial factors. To meet these demands, we have scaled up our HIPIMS developments to deposit coatings on our largest industrial platforms, e.g. the Flexicoat 1500 to address market needs.

We will also provide an outlook on future developments and what can be expected next in the PVD market.

3:20pm **IA2-1-MoA-6 Inorganic Sputtered Coatings to Reduce Snow Friction for Cross-Country Skiing**, ***Pauline Lefebvre [pauline.lefebvre@grenoble-inp.fr]***, SIMAP, Grenoble-INP, CNRS, France; *Fabian Wolfsperger,* WSL Institute for Snow and Avalanche Research SLF, Switzerland; *Jean Herody,* FFS, France; *Matthias Jaggi,* WSL Institute for Snow and Avalanche Research SLF, Switzerland; *Arnaud Mantoux,* SIMaP, CNRS, University Grenoble Alpes, France; *Nicolas Coulmy,* FFS, France; *Pascal Hagenmuller,* Centre d'Etudes de la Neige, CNRM, Météo-France; *Elisabeth Blanquet,* SIMAP, Grenoble-INP, CNRS, France

In cross-country skiing, reducing the friction coefficient between the skis and snow is essential for sportive performance [1]. Fluorinated waxes, i.e. containing perfluoroalkyl (PFA) are known for their hydrophobic properties and were remarkably efficient in wet snow conditions. However, the International Ski and Snowboard Federation (FIS) has banned fluorinated wax since winter 2023/2024 -for health and environmental reasons [2]. Since then, no alternative with equivalent performance has been found. This project aims to develop hard and hydrophobic coatings based on titanium nitride (TiN), aluminum nitride (AlN) and alumina (Al_2O_3) materials directly deposited on ski bases and UHMW polyethylene. The role of coating surface properties and structure in friction is investigated

Thin films were deposited using DC and RF magnetron sputtering. The surface (contact angle, roughness, chemical composition), mechanical and thermal properties of the coatings were investigated. Friction coefficient of coated samples was evaluated on snow with a linear tribometer (speed: 0.1 m/s, displacement: 130 mm, contact pressure: 50 kPa). The tribo-system is therefore a 10cm-long coated ski sliding on controlled man-made snow in a cold-room at 0°C and dry air. Snow with different liquid water content were used for the tests.

Results are encouraging as deposition on ski base is feasible at ambient temperature with adhesive and dense coatings. Coating thicknesses were evaluated by scanning electronic microscopy between 50 and 200 nm depending on process parameters. Chemical analysis with XPS indicates nitride films contain a relative high amount of carbon and oxygen. Coatings, selected for their hydrophobicity and structural properties, were investigated in gliding tests. AlN and Al_2O_3-based coatings presented very high friction coefficient (0.2-0.3). TiN-based coating had the lower friction coefficient with a value of 0.11 on very wet snow, whereas a ski waxed with PFA friction coefficient was measured at 0.072.

To sum up, deposition of sputtered coatings was realized with success and may be a promising technique for preparing competition skis. For winter sport application, titanium nitride seems to be the most promising: it is indeed known for better mechanical properties [3] and lower thermal conductivity [4] which will be further investigated.

References:

[1] Moxnes, J. F. et al, J Sports Med, 4, 127-139 (2013).

[2] Freberg, B. I. et al, Environ Sc & Techno, 44, 7723–7728 (2010).

[3] Glocker, D. A. et al, Bristol, UK: Inst of Phys (1995).

[4] Moraes V. et al, J Appl Phys, 119, 225304 (2016).

4:00pm **IA2-1-MoA-8 Influence of Corrosion on Wear and Brake Particle Emissions of Alumina-Coated and Uncoated Cast Iron Brake Discs**, ***Ran Cai* [cai12r@uwindsor.ca]**, *Xueyuan Nie,* University of Windsor, Canada; *Yezhe Lyu Lyu, Jens Wahlström,* Lund University, Sweden

Hard coatings can be applied to cast iron brake discs to enhance wear and corrosion resistance and reduce brake particle emissions. This study investigated the influence of corrosion on brake particle emissions from cast iron discs through comparison of plasma electrolytic aluminized (PEA)-coated and uncoated surfaces. Six discs were subjected to corrosion in raining-snowy conditions for 24, 48, and 72 hours before undergoing tribological testing using a pin-on-disc tribotester combined with an airborne particle emission measurement system. The counterpart pins were machined from a commercially available low-steel (LS) brake pad. Data of particle concentration, size distribution, and total wear (disc and pad) were collected, while wear tracks, friction transfer layers and worn pad surfaces were analyzed using scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX). The results showed that the degree of corrosion of the uncoated disc increased with time, while the coated discs didn't show any corrosion sign. The corrosion products on the uncoated discs can be cleaned during the early stage of the tribotests where the particle emission was much higher than the later stage. The PEA coating effectively mitigated the effects of corrosion, resulting in significantly lower wear and brake particle emissions compared to uncoated discs. These findings demonstrate the potential of PEA coatings to reduce wear and emissions under winter conditions, offering benefits for environmental sustainability and public health.

4:20pm **IA2-1-MoA-9 The Effect of Mg Addition on the Corrosion Resistance of Two-Step Galvanizing Zn-5Al Coating**, ***Huan-Chang Liang* [hcliang@niu.edu.tw]**, Department of Mechanical and Electro-Mechanical Engineering, National I-Lan University, Taiwan; *Yen-Kai Chen, Chaur-Jeng Wang,* Department of Mechanical Engineering, National Taiwan University of Science and Technology, Taiwan

The atmospheric corrosive substances like Cl^- ions or SO_3^{2-} ions make it easier for soluble zinc salts to form. Consequently, the introduction of more magnesium into the Zn-Al alloy bath enhances the formation of basic zinc salts that are susceptible to environmental corrosion. Zinc-aluminum carbonate hydroxide and aluminum-magnesium carbonate hydroxide are particularly notable corrosion products because of their stable and compact characteristics. Local acidity influences the rate of transformation of zinc hydroxide from zinc oxide, while the formation of $Mg(OH)_2$ mitigates the surface reduction of the galvanizing coating.

Although the Zn-5Al-2Mg coating is effectively produced using the continuous galvanizing process, the maximum coating thickness achieved is 20 µm. Numerous studies present their experimental findings about batch galvanizing zinc alloys. The coating structure is nonuniform, with an enrichment of iron content resulting from the elevated operating temperature, which inhibits the formation of a dense and continuous layer of corrosion products. Consequently, two-step batch galvanizing is utilized to provide a zinc alloy coating of adequate thickness. The initial process involves immersing samples in pure zinc, followed by immersion in zinc alloy. The coating's microstructure consists of an outer layer formed from zinc alloy and internal layers including an iron-aluminum intermetallic compound combined with a eutectic phase.

This study aims to examine the microstructure of two-step batch galvanizing Zn-5Al and Zn-5Al-2Mg coatings on low-carbon steel. The samples are produced by batch galvanizing pure zinc for 10 minutes, followed by batch galvanizing zinc alloy for 2.5 minutes. The corrosion resistance performance of both samples is evaluated by interfacial polarization and impedance matching. The microstructure of two-step galvanizing Zn-5Al and Zn-5Al-Mg consists of four distinct layered structures: a binary (Zn-Al) or ternary (Zn-Al-Mg) eutectic phase layer, a branch-like $FeAl_3$ phase layer, a dense $FeAl_3$ phase layer, and an internal eutectic phase layer. As the zinc content in the coating layer increases, there is a corresponding decrease in the charge transfer impedance (R_{ct}). This behavior is ascribed to the area fraction of the $FeAl_3$ phase in conjunction with the eutectic phase. The addition of magnesium into the zinc alloy bath enhances the R_{ct} of the entire coating layer. The advantageous effect arises from the disparity in corrosion potential between magnesium and zinc. Magnesium functions as a sacrificial anode for the Zn-rich phase, hence enhancing the effectiveness of cathodic protection.

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