Tuesday Afternoon, May 13, 2025

Tribology and Mechanics of Coatings and Surfaces Room Palm 5-6 - Session MC2-1-TuA

Mechanical Properties and Adhesion I

Moderator: Alice Lassnig, Austrian Academy of Sciences, Austria

1:40pm MC2-1-TuA-1 Nanoscale Interface Engineering for Thin Films on Polymer Substrates, Barbara Putz [barbara.putz@empa.ch], EMPA (Swiss Federal Laboratories for Materials Science and Technology), Switzerland

Atomic layer deposition (ALD) holds enormous potential to design interfaces, due to the unique way in which a material is built in an atomic layer-by-layer fashion. When combined with other thin film techniques, such as magnetron sputtering (PVD), without breaking vacuum, the layer-by-layer nature of ALD can be harvested to design (sub)nanoscale interface architectures. An interesting area for this combined deposition are metal-polymer interfaces, where thin amorphous interlayers (IL, 5 nm thick) between metal film and polymer substrate favour strong and stable interfaces [1-3]. Until now, interlayer formation is governed by the film/substrate chemistry and deposition method, preventing high interface quality for the majority of material combinations and fabrication routes. Since ultrathin ALD layers uniquely resemble the reported interlayer in structure and chemistry, interlayer formation can, for the first time, be mimicked artificially to clarify the role of these structures in thin film delamination.

Through a combined ALD/PVD setup, we fabricate and study Al thin films (150 nm) with different ALD interlayer thicknesses (Al $_2$ O $_3$ + H, 0.12 - 25 nm) on a polyimide substrate. Mechanical properties are measured via uni- and equi-biaxial tensile loading [4] with in-situ X-ray diffraction and electrical resistivity measurements from the evolution of Al film stress, width of the Al diffraction peak and electrical resistivity as a function of IL thickness and applied strain. Adhesion energy between metal film and polymer substrate is calculated using the tensile induced delamination method.

In our study, differences in the system's mechanical behaviour (yield strength, crack onset strain) are found to be driven by the microstructure of the metallic Al layer (film thickness and grain size), while the crack propagation (electrical failure strain) and adhesive performance (buckle density) is dominated by the interface structure. Significant embrittlement and fracture is only observed for thick interlayers (>= 25 nm).

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- [2] Putz, B. et al. Surf. Coat. Technol. 332, 368-375 (2017)
- [3] S. Oh. Et al., Scripta Materialia 65 (2011) 456-459

2:20pm MC2-1-TuA-3 Trilayer Fracture and Adhesion Investigated with in-Situ Synchrotron Radiation, Megan J. Cordill [megan.cordill@oeaw.ac.at], Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Austria; Shuhel Altaf Husain, Université Sorbonne Paris Nord, France; Claus O.W. Trost, Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Austria; Damien Faurie, Université Sorbonne Paris Nord, France; Pierre O. Renault, University of Poitiers, Pprime Institute, France

Flexible and wearable electronics use multiple metal films on polymer substrates to achieve functionality where the resistance to through thickness fracture and the adhesion to the polymer substrates determines device performance. Commonly, flexible material systems are made of layers ductile metals of copper or aluminum as the conducting layers with more brittle molybdenum and chromium used as interlayers to improve adhesion to the polymer substrate or as protective capping layers. In this work, in-situ uniaxial tensile straining was used to investigate the fracture and delamination behavior of brittle-ductile-brittle trilayers. The method uses uniaxial straining to cause fracture of the film system perpendicular to the tensile loading direction and film delamination parallel to the tensile loading direction, which allows the adhesion energy to be evaluated. Experiments on the differently layered samples, namely Mo-Cu-Cr and Nb-Cu-Mo, were performed with in-situ resistance measurements and X-ray diffraction (XRD). Combined with post-straining confocal laser scanning microscopy, XRD provided the film stress evolution simultaneously in every layer to understand fracture of trilayer systems and how adhesion can be measured using tensile induced delamination. The main aspects presented will be adhesion energy along with the stress evolution under uniaxial tensile loading of the various trilayer architectures. Results indicated that the position of the Mo layer can influence the fracture behavior. It was also observed that only the presence of a brittle layer, rather than the position (interface layer vs. top layer), aids delamination in trilayers. Compared to single layer films of similar thickness, no significant change in the calculated adhesion energy of the same trilayer interfaces was found.

2:40pm MC2-1-TuA-4 The Model to Explain the Origin of Residual Thin Film Stress, Tong Su [tong_su@brown.edu], Eric Chason, Brown University, IISA

Residual stress has been a long-standing problem in thin film deposition, and it is critical to the adhesion and physical properties of their applications. In previous works, we have studied the mechanisms and used modeling to explain the stress evolution in the post-coalescence stage (typically 50 nm ~ 400 nm) and steady state (>400 nm or when the stress does not change significantly). The early stage of growth (<50 nm) is not as well studied as the others and vet this state is important to the origin of the thin film stress. Here we present a model to explain the behavior of residual stress in the early stage with the assumption that the deposited particles form hemisphere islands on the substrate. The model is applied to analyze stress measurements of e-beam evaporated Ag and Ni from the wafer curvature measurements. The results suggest that the end of the coalescence stage may not be sufficient to explain the occurrence of the tensile peak in the early state. Rather, the balance between tensile and compressive stress mechanisms as the grain boundary is formed between islands needs to be considered.

3:00pm MC2-1-TuA-5 Novel Approach for Scratch Analysis of Ductile Metallic Layers on Fragile Substrates, Mohammad Arab Pour Yazdi, Pavel Sedmak, Anton Paar TriTec SA, Switzerland; Parth Kotak, Anton Paar USA; Jiri Nohava, Anton Paar TriTec SA, Switzerland; Mark Haase [mark.haase@anton-paar.com], Anton Paar, USA

In the electronics and semiconductor industries, there is a growing demand for precise characterization of adhesion properties in soft metallic multilayers on fragile substrates, such as semiconductor wafers and glass. Consequently, nondestructive testing methods have become essential to prevent damage to these sensitive substrates during testing. Conductive metallic layers including gold (Au), platinum (Pt), copper (Cu), and silver (Ag) are critical for microchip pathways; however, their ductility poses challenges for adhesion testing on brittle substrates. Traditional nanoscratch methods, which use sphero-conical indenters, rapidly traverse these soft layers and exert significant stress on the fragile substrates. This often results in substrate failure rather than yielding valuable insights into the interfacial adhesion of the layers.

In this study, we introduce a novel scratch testing method specifically designed for soft metallic layers or multilayers on fragile substrates. This approach employs a micro wedge blade indenter, rather than the conventional spheroconical indenter, along with a two-axis tilt stage sample holder, enhancing precision and reducing substrate damage to yield more reliable adhesion measurements. This method is particularly suited for ductile metallic coatings deposited via PVD, CVD, and ALD, providing a robust solution for accurately assessing the adhesion properties of soft metallic coatings on sensitive substrates.

Keywords: Ductile coatings; Fragile substrates; Adhesion testing; Wedge blade indenter; Nanoscratch testing.

4:00pm MC2-1-TuA-8 The Comparison in Microstructure and Mechanical Properties of MoN Films Deposited by RFMS and HiPIMS Techniques, *Chi-Yueh Chang [w6208asx@gmail.com]*, National United University, Taiwan

The MoN films are gathering increasing attentions for their highperformance characteristics, making the production of high-quality MoN films an important research focus. In this study, Mo-N thin films are coated using radio frequency magnetron sputtering, RFMS, and high intensity power impulse magnetron sputtering, HiPIMS, techniques. The input power and Ar/N2 ratio are adjusted from 150 to 200W and 15/5 to 18/2 sccm/sccm to control the microstructure. The duty cycle of the HiPIMS from 4 to 10% is also manipulated to trigger higher peak power density and current. A columnar structure feature was observed across all thin films. Nevertheless, the phase of the Mo-N changes under different parameters. Through RFMS, as the Ar/N₂ ratio was raised from 15/5 to 18/2 sccm at 150 W input power, a significant evolution of major Mo₂N to MoN phase was observed. With higher peak current and power density through HiPIMS deposition, a multiple phase feature with decreased grain size of Mo-N phases were discovered. The microhardness, elastic modulus, wear resistance and indentation cracking behavior were investigated. The correlation between microstructure evolution and the mechanical properties were also discussed.

Keywords:RefractoryThin film coatingsMoNHipims

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4:20pm MC2-1-TuA-9 Quantitative 3D FIB-SEM Characterization of Single Cu Particle Impacts for Cold Spray Applications, Veera Panova [vpanova@mit.edu], Massachusetts Institute of Technology, USA; Christopher Schuh, Northwestern University, USA

Cold spray is a solid-state additive manufacturing process that produces coatings and standalone parts by accelerating micron-sized metallic particles to supersonic velocities. Upon impact, the particles and substrate undergo plastic deformation, surface oxide layers get disrupted, and direct particle-substrate contact is achieved to attain metallurgical bonding. Our recent works take advantage of the Laser-Induced Particle Impact Test (LIPIT) to produce single microparticle impacts under carefully controlled conditions, providing a unit-process understanding of cold spray physics. Each launched particle is well-characterized: its size, morphology, microstructure, velocity, and in-flight behavior are known. We then analyze impact sites using focused ion beam-scanning electron microscopy (FIB-SEM) to study multiple aspects of the impact event: bonding at particlesubstrate and particle-particle interfaces, deformation at high strain rates, and microstructural evolution. The major advantage of this approach is that it is tomographic, providing direct 3D observations of the interfaces, as well as quantitative measurements of the bonded area and microstructural changes around the impact site.

This talk will review several observations that 3D tomography of the impact sites reveals about structure development in cold spray. First, we observe generally non-symmetrical bonding at the particle-substrate interface and conclude that bonding takes place top-down; regions experiencing high strain bond first. These insights conform to a model for particle-substrate bonding through oxide-layer rarefication and provide guidelines for how to optimize processing parameters to produce well-bonded cold spray coatings. Second, our microstructural observations reveal limiting conditions for the development of recrystallization structures. Such information speaks to the development and dissipation of adiabatic heat upon impact.

4:40pm MC2-1-TuA-10 Mechanical Properties and Deformation Mechanisms of Metallic Thin Films Synthetized by Pulsed Laser Deposition, Francesco Bignoli, Davide Vacirca, Philippe Djemia, Laboratoire des Sciences des Procédés et des Matériaux (LSPM) – CNRS, France; Andrea Li Bassi, Department of Energy, Politecnico di Milano, Italy; James Paul Best, Gerhard Dehm, Max-Planck Institut für Eisenforschung, Germany; Matteo Ghidelli [matteo.ghidelli@lspm.cnrs.fr], Laboratoire des Sciences des Procédés et des Matériaux (LSPM) – CNRS, France

The ongoing trend toward miniaturization in device components across key technologies demands the synthesis of high-performance nanostructured films with exceptional combination mechanical properties such as high yield strength and plasticity which, however, are mutually exclusive. In order to overcome such trade off, it is crucial to control the atomic composition and the microstructure, going beyond currently nanoengineering design approaches for thin films. One main limitation arises from conventional thin film deposition techniques (sputtering) with limited possibility to fabricate novel microstructures such as with ultrafine grains or nanoscale laminates alternating layers of different compositions and phases with intrinsic dimensions on the order of a few nanometers. Such features could induce mechanical size effects, influencing deformation mechanisms and enabling highly tunable and enhanced mechanical properties.

Here, I will show the potential of Pulsed Laser Deposition (PLD) as a novel technique to synthetize advanced metallic thin films, reporting the fabrication of a variety of microstructures with tailored composition and nanoscale features including compact, nanogranular and crystal/glass ultrafine nanolaminates and focusing on the deformation behavior and mechanical properties.

First, I will focus on the on the fabrication of thin film metallic glasses with different composition ZrCu, ZrCuAl (with also O addition) and controlled microstructure, compact and nanogranular [1]. The mechanical characterization with optoacoustic techniques, nanoindentation and *in situ* SEM micropillar compression reports large and tailored mechanical properties, above sputter-deposited counterparts, reaching ultimate yield strength (>4 GPa) and ductility (>15 %) for ZrCuAl/O films. Then, I will show the fabrication of ultrafine glass/crystal (ZrCu/Al) nanolaminates with high and tunable density of interfaces (nanolayer thickness <5 nm), reporting shear bands blocking and homogenous deformation, in combination with large plasticity (> 10%) and yield strength (>3.4 GPa) [2].

Lastly, I will focus on the PLD synthesis of CoCrCuFeNi crystalline high entropy alloys showing unique microstructure and ultrafine grains (≈10

nm), triggering Hall-Petch strengthening resulting in high hardness (≈10.5 GPa) and yield strength (1.9 GPa) significantly above sputter-deposited counterparts, while retaining large plastic deformability (30%) [3].

- [1] M. Ghidelli et al., Acta Mater., 213, 116955, 2021.
- [2] F. Bignoli et al., ACS Appl. Mater. Interfaces, 16, 27, 35686-96, 2024.
- [3] D. Vacirca et al., Submitted to Acta Mater, 2024.

5:00pm MC2-1-TuA-11 The Forgotten Method: Coatings Mechanical Properties Calculated According to ISO Standard 14577, Esteban Broitman [ebroitm@hotmail.com], EDB Engineering Consulting, France

When an indenter penetrates the surface of a film deposited onto a substrate, the mechanical response of the film will be influenced by the mechanical properties of the substrate, according to its penetration depth h and the film thickness *t*. As the depth of penetration h increases, more of the mechanical contribution will come from the substrate.

From the first work published by H. Bückle in 1959 for microindentations, there have been many theoretical and experimental published research works trying to show how the hardness and elastic modulus of the coatings should be calculated in order to avoid any influence of the substrate [1].

ISO Standard 14577 "Metallic Materials—Instrumented Indentation Test for Hardness and Materials Parameters" published for first time in 2002, was written to make some order in the way to use nanoindenters. The standard included originally 3 parts: Part 1: Test method; Part 2: Verification and calibration of testing machines; and Part 3: Calibration of reference blocks. Some years later, the standard included a new Part 4: Test method for metallic and non-metallic coatings. This section of the Standard contains a method that has been ignored by most of researchers.

In this presentation, we will review the four parts of ISO standard 14577. In particular, we will analyze the simple experimental methodology established in Part 4 that, in most of cases, gives the correct values for hardness and elastic modulus, independently of the coating/substrate system.

[1] E. Broitman, Indentation Hardness Measurements at Macro-, Micro-, and Nanoscale: A Critical Overview. Tribol. Lett. 65 (2017) 23.

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Tribology and Mechanics of Coatings and Surfaces Room Palm 5-6 - Session MC2-2-WeM

Mechanical Properties and Adhesion II

Moderator: Chia-Lin Li, Ming Chi University of Technology, Taiwan

8:00am MC2-2-WeM-1 Adhesion, Delamination and Cracking of Thermal Spray Coatings: Understanding Critical Phenomena During Processing and Service, Sanjay Sampath [sanjay.sampath@stonybrook.edu], Stony Brook University, USA

INVITED

The efficacy of coatings in engineering applications rely on their ability to be well bonded to the underlying substrate. Many factors govern this adhesion including deposition materials, substrate materials, substrate attributes, surface chemistry, processing conditions, thickness, build rate, mismatch between the coating and substrate etc. Methods to measure adhesion in present day is largely phenomenological with "go/no-go" agenda. Of importance is that today's measures of adhesion strength may not be appropriate for coatings which are largely brittle, where cracking is a predominant mode of failure representing a toughness problem rather than strength consideration. Furthermore, even well bonded coatings can delaminate during service where compounding effects of service load can superpose to accentuate the interfacial stresses. Thus, understanding these phenomena is critical. The debonding of the interface is driven by energy dissipation. In situation where bonding is strong, an alternative energy release mechanism is cracking of the coating. When harnessed they provide a pathway to build strain-tolerant vertically cracked coating with implications for novel design and manufacturing of thermo-structural coatings. In many instances, the factors of cracking and delamination compete. This is dependent on adhesion and microstructure. In this presentation, the above attributes are critically discussed through phenomenological and quantitative strategies.

8:40am MC2-2-WeM-3 A Study on the Surface Morphology and Tribological Behavior of Hydrided Zircaloy, Jun Xian Lin [linst214200@gmail.com], Kuan-Che Lan, National Tsing Hua University, Taiwan

The integrity of used nuclear fuel claddings is one of the keys to assess the safety margin during interim dry storage. Nuclear fuel claddings made of zirconium alloys have been widely applied in commercial nuclear reactors such as boiling and pressurized water reactors. The accumulation of hydrogen in the form of zirconium hydride which could deteriorate the integrity of used nuclear fuel claddings during interim dry storage is one of critical concerns intrinsically. Besides, existence of hydride in zirconium alloys could weaken the tribological resistance of the cladding materials during the loading and transportation procedures of used fuel prior to a long-term dry storage and hurt the integrity externally. A thoroughly understanding of about the microstructure and tribological behavior of zirconium alloy with hydrides will improve the reliability of evaluation on the integrity of used fuel cladding during interim dry storage. The objective is to study the influence of zirconium hydride on the tribological resistance. Scratch tests were conducted on as-hydrided Zircaloy-4 plate using a scratch tester to determine the minimum load causing cracks and to analyze the morphology of surface cracks. Additionally, a pin-on-disc test was conducted to assess the wear resistance, followed by SEM analysis over the damaged surface to observe the effect of hydrogen permeation on the tribological behavior of the Zircaloy-4.

9:00am MC2-2-WeM-4 Effects of Stored Elastic Energy and Stress Gradients on the Tribological Behavior of TiN Coatings on D2 Steel, I-Sheng Ting [gary820902@yahoo.com.tw], Jia-Hong Huang, National Tsing Hua University, Taiwan

Residual stress is one of the most pivotal issues in protective hard coatings deposited by physical vapor deposition methods. It is generally acknowledged that low residual stress is beneficial for prolonging the lifespan of hard coatings. In our previous studies [1,2], a Ti interlayer was added to alleviate the residual stress of TiZrN coating on D2 steel, thereby improving its wear resistance. An energy-based hypothesis was proposed to explain the enhancement in wear resistance [2], where by lowering the stored elastic energy (Gs) in the TiZrN coating, the margin for reaching the fracture toughness (Gc) was extended, indicating that the coating could endure more external loading. However, the energy-based perspective neglected the effect of stress gradient that significantly affects the propagation of cracks in coatings. This study aimed to measure the stored elastic energy and energy gradients of TiN coating on D2 steel and evaluate

the effect of gradients on the tribological behavior. TiN coatings were deposited on D2 steel and Si substrates using DC unbalanced magnetron sputtering, where the stress gradient of TiN coating was controlled by adjusting the working pressure during deposition. The average stress of the TiN coating was determined using the average X-ray strain (AXS) combined with nanoindentation methods [3-5], and the stress gradient was acquired by changing the X-ray incident grazing angles. The adhesion and wear resistance of the TiN coatings on D2 steel were respectively evaluated using scratch test and pin-on-disk wear test. Through the adjustment of working pressure during deposition, it is feasible to control the tribological behavior of a hard coating by tuning the distribution of stored elastic energy and stress gradients.

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9:20am MC2-2-WeM-5 Adhesion at the Glass/Metal interface probed by Colored Picosecond Acoustics, Arnaud Devos [arnaud.devos@iemn.fr], IEMN, France

Glass is a common material already employed in everyday applications, which has gainedconsiderable interest for electronic components, due to its attractive electrical, physical, andchemical properties, as well as its prospects for a cost-efficient solution. Adhesion of thin metal film on glass is especially critical and bonding between glass and metal can broaden the applications of glass in many industrial areas. A large number of methods have been developed to characterize the adhesion of a thin film to a substrate. Acoustic waves and especially ultra-high frequency acoustic waves are also sensitive to adhesion defects as they affect the way acoustic waves are transmitted and reflected at the interface concerned. At a poor interface, acoustic waves are much more reflected than expected and therefore much less transmitted. In this work, we use picosecond acoustics for measuring the metal film thickness and the acoustic transmission coefficient at the interface with a glass substrate. Picosecond acoustics is a ultrafast laser technique that implements a nanoscale pulse-echo technique[1]. A femtosecond optical pulse excites a short acoustic pulse inside the sample and another optical pulse is used to monitor acoustic propagation and reflections. We show that we can take advantage of the laser tunability to improve the measurement of adhesion between metal and glass: by making picosecond acoustic measurements at different wavelengths (spectroscopy), we observe very sensitive changes in the photo-acoustic response which can be used to improve measurement

References:[1] A. Devos, Ultrasonics 56, pp. 90-97 (2015) DOI 10.1016/j.ultras.2014.02.009

9:40am MC2-2-WeM-6 The Mechanical and Tribological Performance of (V,Mo)N Coatings Deposited by Magnetron Sputtering, Yuqun Feng, Jia-Hong Huang [jhhuang@ess.nthu.edu.tw], National Tsing Hua University, Taiwan

The wear resistance of transition metal nitrides (TMeNs) can be enhanced by introducing self-lubricating oxide forming alloy elements, such as V and Mo. However, TMeNs are usually brittle under dynamic loading conditions. (V,Mo)N is a recently developed material for wear-resistant coatings due to its high fracture toughness. The objective of this study was to evaluate the mechanical and tribological properties of single-phase (V,Mo)N coatings. (V,Mo)N coatings with different N/metal ratios were deposited on AISI D2 steel substrates using direct current unbalanced magnetron sputtering (dc-UBMS) and high power pulsed magnetron sputtering (HPPMS). The results showed that the coatings deposited on steel substrates have higher N/metal ratio and (200)-preferred orientation than those on Si substrates. This may be attributed to the higher electrical conductivity of the steel substrate, leading to more intense ion bombardment that delivers more energy in forming N-metal bonding and enhances the channeling effect. The hardness of the coatings increases with decreasing N/metal ratio. Additionally, the coatings deposited by HPPMS on steel substrates have lower residual stress than those by dc-UBMS. This may be due to the stress induced by the power cycle being relieved by plastic deformation of the steel substrate. All (V,Mo)N coatings show a very low wear rate ranging

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from 1.1×10^{-7} to 4.0×10^{-7} mm $^3N^1m^1$ at room temperature. As temperature increases to 500 °C and above, the wear resistance of the (V,Mo)N coatings significantly decreases, while low friction coefficients are maintained by the formation of self-lubricating V- and Mo-oxides. All coatings remain intact after 150k impact fatigue test, even when the deformation depth is larger than the coating thickness, implying the remarkable toughness of the (V,Mo)N coatings. In contrast, the coatings deposited using dc-UBMS have the worst impact fatigue resistance, which may be related to their lower fracture toughness.

Tribology and Mechanics of Coatings and Surfaces Room Town & Country C - Session MC3-1-WeM

Tribology of Coatings and Surfaces for Industrial Applications I

Moderators: Stephan Tremmel, University of Bayreuth, Germany, Martin Welters, KCS Europe GmbH, Germany

9:00am MC3-1-WeM-4 Cyclic and Randomized Micro-Impact Tests of Coatings for Erosion Protection: Role of Multilayer Structure in Providing Damage Tolerance, Ben Beake [ben@micromaterials.co.uk], Micro Materials Ltd, UK; Daniel Tobola, Lukasiewicz Research Network, Krakow Institute of Technology, Poland; Luksaz Maj, Institute of Metallurgy and Materials Science of Polish Academy of Sciences, Krakow, Poland; Tomasz Liskiewicz, Manchester Metropolitan University, UK; Puneet Chandran, Lukasiewicz Research Network, Krakow Institute of Technology, Poland Coating systems for applications in machining and forming tools, and in

applications where they are subject to solid particle erosive wear, are subject to high loads which can result in high wear and premature failure. To aid the design of coating systems to mitigate this with improved surface fatigue resistance, cyclic micro-impact tests have been performed on three hard multilayered coatings (TiN/TiCrN/TiN, TiN/TiCrN/10x(TiN/CrN)/TiN and 25x(Cr/CrN)) deposited by arc evaporation onto hardened tool steel and results compared to a monolayer TiN reference. To more closely replicate the statistical, and apparently stochastic, distribution of multiple impacts that occur in solid particle erosion randomized micro-impact tests were performed where multiple impacts occur with controlled energy at different (chosen) locations on the coating surface. The cyclic and randomized impact tests were both performed using a multi-sensing approach where the depth and dissipated energy were monitored for every impact improving detection of the onset of severe wear. The multilayered TiN-based coatings were more prone to chipping than the monolayer TiN in the cyclic and randomized tests. Although the 25x(Cr/CrN) coating was susceptible to radial cracking and cracking within impact craters this localized cracking relieved the impact-induced stresses and minimized the chipping failure found on the other coatings. SEM and TEM imaging has been used to investigate the impact damage phenomena.

9:20am MC3-1-WeM-5 Effect of Bias Voltage and Temperature on the Structural and Tribo-Mechanical Properties of Chemically Complex Tisibcn Nanocomposites, Wolfgang Tillmann, Julia Urbanczyk [julia.urbanczyk@tu-dortmund.de], TU Dortmund University, Germany; Alexander Thewes, TU Braunschweig University, Germany; Nelson Filipe Lopes Dias, TU Dortmund University, Germany

TiSiBCN thin films show promising properties for applications at elevated temperatures due to improved thermal stability and oxidation resistance, as well as friction-reducing characteristics. While previous studies investigated mainly the effect of the chemical composition on the thin film properties, it remains unclear how deposition parameters, such as the bias voltage and the heating power, affect the structural and tribo-mechanical properties of TiSiBCN. For this reason, the effect of the bias voltage and heating power on magnetron-sputtered TiSiBCN nanocomposites with different chemical compositions was analyzed. In the first line of investigation, the bias voltage was varied from -100, -150, and -200 V, and in the second line, the heating power was set to 2, 5, and 8 kW.

The chemical composition remains nearly unaffected by the heating power, while the bias voltage has a slight effect on the quantity of the elements. X-ray diffraction (XRD) analysis revealed a polycrystalline structure with randomly oriented crystallites, characterized by different peak shifts depending on the chemical composition. Identified crystalline phases include TiN, TiC, TiB, and TiB₂, coexisting with various amorphous phases. Transmission electron microscopy (TEM) images reveal a nanocomposite structure and changes in microstructure, such as crystallite refinement with higher bias voltage or growth, as well as further self-assembly with higher

deposition temperatures, depending on the chemical composition and initial phase structure. An increased bias voltage induces residual stresses while the hardness tends to decrease. With higher heating power, internal stresses are released and the hardness increases up to 41 GPa. To explore the application potential of the TiSiBCN thin films for forming processes of aluminum alloys, the tribological behavior was evaluated against AW-6060 in tribometer tests, highlighting TiSiBCN as a promising protective coating.

9:40am MC3-1-WeM-6 Lubrication Mechanism of CrAIN+MoWS Coatings in Gear Contacts under Dry Rolling-Sliding Conditions, Kirsten Bobzin, Christian Kalscheuer, Max Philip Möbius, Marta Miranda Marti [marti@iot.rwth-aachen.de], Surface Engineering Institute - RWTH Aachen University, Germany

The use of liquid lubricants for wear and friction reduction in geared transmissions is well established. However, in applications like the food industry, liquid lubricants are undesirable due to contamination risks. A promising alternative involves applying a wear-resistant CrAIN coating incorporated with solid lubricant components, such as molybdenum, tungsten and sulfur. Previous studies demonstrated the functionality of graded CrAIN+MoWS coatings, analyzing the lubrication mechanism on flat samples using pin-on-disc method. Further studies extended this analysis to gear applications, where the coating reduced friction and wear by 88 % compared to uncoated contacts.

In this study the lubrication mechanism of PVD deposited graded CrAIN+MoWS on gears was analyzed. The coated wheels were tested against uncoated pinions under varying Hertzian pressure at pitch point, with p_{H1} = 589 N/mm² and p_{H2} = 1.723 N/mm², and circumferential speed v_{t1} = 2 m/s and v_{t2} = 8,3 m/s. After tribological testing, the gear tooth surfaces were examined using confocal laser scanning microscopy (CLSM) and energy-dispersive X-ray spectroscopy (EDX) to determine the coating distribution. Raman spectroscopy was employed to analyze the possible formation of the solid lubricant MoS2 and WS2 phases, as well as other friction-reducing oxides. At lower Hertzian pressures, the triboactive elements on the wheel tooth flank are effectively consumed, leading to a friction reduction compared to uncoated gear contacts. On the wheel tooth faces, the triboactive elements remain present and are identified through Raman spectroscopy as MoS₂, which could further contribute to friction reduction. On the corresponding uncoated pinions, traces of Mo. W. and S are detected, confirming the effective transfer mechanism of CrAIN+MoWS coatings in gear contacts at lower Hertzian pressure. At higher Hertzian pressures and high circumferential speeds, traces of MoS₂ are observed on the wheel tooth face, indicating the coating consumption to reduce friction and demonstrating the effectiveness of the coating under extreme testing conditions, which expand the gear's lifespan compared to uncoated gear contacts.

The results demonstrate the lubrication mechanism of the CrAlN+MoWS coating in gear contact. MoS_2 is generated at the gear contact, even under low Hertzian pressure, and is efficiently utilized within the contact zone to ensure a friction reduction. At higher speeds, these triboactive elements remain effective, continuing to enhance lubrication and reduce wear within the gear contact when compared to uncoated gears.

11:00am MC3-1-WeM-10 Wear Protection via Triboactive CrAIMON Coatings in Chain Drives, Kirsten Bobzin, Christian Kalscheuer, Max Philip Möbius [moebius@iot.rwth-aachen.de], Surface Engineering Institute - RWTH Aachen University, Germany; Martin Rank, Oliver Koch, Institute of Machine Elements, Gears and Tribology - RPTU Kaiserslautern-Landau, Germany

Within chain drives, critical wear occurs between the chain pin and chain bush, leading to chain elongation. This determines the service life of a chain. Hard coatings deposited by physical vapor deposition (PVD), such as CrAIN, can effectively reduce wear. However, coating the inner surfaces of chain bushes presents economic and technological challenges. A promising alternative is the use of triboactive CrAIMON coatings, which interact with lubricants and their additives to form protective tribofilms. These tribofilms can transfer to uncoated chain bushes, providing essential wear protection.

In this study, three chains were assembled using uncoated, CrAIN and CrAIMoN coated pins. These chains were then tested on a chain drive test bench. All chains were lubricated with grease containing sulfur additives. Analyses of the as-coated chain pins included geometry, surface roughness, coating thickness, coating morphology and compound adhesion. The chains underwent testing under medium load conditions corresponding to a power transmission of $P_M = 2.3$ kW and high load conditions corresponding to $P_H = 9.5$ kW. Wear was monitored through periodic measurements of chain elongation to determine wear rates over time. Upon completion of

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testing, both chain pins and bushes were analyzed for visual appearance changes, wear volume, surface topography, and remaining coating thickness. Under medium load conditions, CrAlMoN coated chains exhibited slightly higher wear rates compared to reference systems. However, under high load conditions, CrAlMoN coated chains demonstrated the lowest wear rates among all tested configurations. Notably, wear distribution between the chain pin and bush was more uniform in CrAlMoN coated systems compared to others where higher wear predominantly affected uncoated bushes.

This observation suggests that the formation and transfer of protective tribofilms in CrAlMoN systems contribute significantly to enhanced wear resistance under high stress conditions. Analysis after high-load testing revealed that CrAlMoN coated pins retained substantial coating thickness within the wear areas of the pin.The findings indicate that triboactive CrAlMoN coatings hold considerable promise for reducing wear in high-performance chain drives by forming protective tribofilms during tribological operation that can be transferred to uncoated chain bushings.

11:20am MC3-1-WeM-11 Tribological Contact Formation on PVD-Coated Tools, Aljaz Drnovsek [aljaz.drnovsek@ijs.si], Peter Panjan, Matjaž Panjan, Miha Čekada, Jožef Stefan Institute, Slovenia INVITED

Tools surface topography changes dramatically after PVD coating deposition. Various topographical imperfections on the coating surface can negatively impact the quality of the coating and, in some cases, cause the failure of the coating. The imperfections in coated forming tools initiated over a decade of research into the phenomena associated with coating surfaces, particularly the growth defects.

I will present results related to the formation of the coating topography and how it depends on factors such as substrate material, ion etching, and deposition processes. The topographical features of the coating significantly influence oxidation, corrosion, and especially the tribological behavior of PVD coatings.

The influence of the coated surface on the formation of a tribological contact has been the focus of several studies, as the contact area between two sliding bodies is not constant with time. Initially, only the asperities which appear as growth defects are in real contact with the counter body. Under load, these asperities can fracture, spall, and produce small particles. The real contact area is increasing sharply before it stabilizes. In terms of friction, we recognize this behavior as the running-in period. The coefficient of friction increases in this period until it reaches a steady state value. It is still poorly understood how this transition from the run-in to the steady state friction occurs and, more importantly, how the growth defects affect the tribological performance. The role of defects in the formation of the tribological contact changes depending on counter body materials and operating temperature. The latter was studied recently. The results indicate that in the case of the TiAIN coating, the highest wear was measured during the room temperature test. Conversely, the wear during the running-in phase and steady-state friction were low at elevated temperatures initially. but as the temperature increased, the wear rate rose, which can be attributed to increased tribological oxidation and fatigue.

The growth defects on the coating surface played a significant role in the friction and wear behavior, as they were a primary source of wear particles and the first spots of oxidation on the coating. The measurements suggest that the running-in phase depends mainly on the asperities density at room temperature tests. In contrast, at high temperatures, they attributed to the formation of a stable tribological oxide layer in the wear track, which elongates the running-in period and protects the coating underneath.

12:00pm MC3-1-WeM-13 Effect of Transition Metals (Nb, V, and Ta) Doping on the High-Temperature Mechanical and Tribological Properties of CrYN Coatings, Gokhan Gulten, Banu YAYLALI, Mustafa YESILYURT, Yasar TOTIK, Atatürk University, Turkey; Justyna Kulczyk Malecka, Peter Kelly, Manchester Metropolitan University, U.K.; Ihsan Efeoglu [iefeoglu@atauni.edu.tr], Atatürk University, Turkey

This study aims to develop a high temperature wear resistant coating for AISI 316L. As a functional coating, CrYN coatings with added niobium, tantalum, and vanadium (a-C:H:Nb/Ta/V) were deposited using a closed-field unbalanced magnetron sputtering (CFUBMS) system. The Taguchi L9 orthogonal array approach was used to test and systematically change a variety of parameters in order to achieve the optimal coating properties. The microstructural properties of the coatings were examined using a scanning electron microscope (SEM), while X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS) analysis were conducted to determine crystallographic and surface chemistry properties, providing a detailed understanding of the coating structure. Nanoindentation tests were

performed to determine mechanical properties, yielding precise measurements of hardness and elasticity. The adhesion of the coatings was measured through scratch tests at varying temperatures (400, 600, and 800 °C) and room temperature. The tribological characteristics of the a-C:H:Nb/Ta/V coatings were assessed using a high-temperature pin-on-disc tribometer, examining their wear resistance and frictional behavior under ambient air and at varying temperatures (400, 600, and 800 °C). These comprehensive analyses reveal the potential of the a-C:H:Nb/Ta/V coatings for applications requiring enhanced surface properties, offering superior tribological performance across different temperature conditions.

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Tribology and Mechanics of Coatings and Surfaces Room Town & Country C - Session MC3-2-WeA

Tribology of Coatings and Surfaces for Industrial Applications II

Moderator: Dominic Stangier, Oerlikon Balzers Coating Germany GmbH, Germany

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

As electric vehicles (EVs) become more widespread, managing electrical current effects on friction and wear in moving components is crucial for enhancing durability and efficiency. Diamond-like carbon (DLC) coatings, known for their low friction and insulating properties, show potential in these applications. This study investigates the tribological characteristics of two types of tetrahedral amorphous carbon (ta-C) coatings -soft (51 GPa) and hard (69 GPa)- on high-speed steel (HSS) substrates under the electrical current application. The soft ta-C coating was deposited at 150 °C, while the hard ta-C coating was deposited at room temperature with a -100 V substrate bias, both using filtered cathodic vacuum arc (FCVA) with a Ti interlayer deposited by magnetron sputtering. The average surface roughness (R_a) values were 17.1 ± 0.3 nm for the soft ta-C coating and 20.3 ± 0.9 nm for the hard ta-C coating. Friction and wear resistance were evaluated using a modified ball-on-disk tribometer with an AISI 52100 steel counterface, under electrical currents from 0 to 1500 mA. Under nonelectrified conditions, both hard and soft ta-C coatings displayed low wear rates of 4.5 and 5.27 × 10⁻⁷ mm³/m.N, respectively. With applied electrical currents, however, notable differences emerged. The hard ta-C coating demonstrated coefficient of friction (COF) values ranging from 0.11 to 0.44 under electrical currents between 0 and 500 mA. In comparison, the soft ta-C coating exhibited lower COF values, ranging from 0.11 to 0.29, across a broader current range of up to 1500 mA. The wear rate of the hard ta-C coating increased significantly to $1.6 \times 10^{-5} \text{ mm}^3/\text{m} \cdot \text{N}$ at 300 mA, whereas the soft ta-C coating maintained a much lower wear rate of 1.05 × 10⁻⁶ mm³/m·N at the same current and reached only 6.17×10^{-6} mm³/m·N at 1200 mA. These results indicate that the electrical current carrying tribological performance of ta-C coatings on HSS substrates can be tailored by heat treatment to enhance their response. Raman spectroscopy and electron microscopy are utilized to delineate the mechanisms underlying these structural changes and will be presented at the conference.

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

Amorphous carbon (a-C) coatings, composed of sp² and sp³ hybridizations of carbon, may enhance the surface properties of materials. These coatings are commonly used as solid lubricants, improving tribological performance by forming a tribolayer that reduces the coefficient of friction by graphitization. In many systems, a-C coatings offer the potential to lower frictional energy losses and wear, improving efficiency and durability. Specific phenomena are anticipated for electric vehicles (EVs), since, from one side, electric current can affect surface wear in electrified systems by promoting accelerated oxidation or arc formation. On the other hand, electrical current flowing through an a-C coated contact can induce carbon crystallization, benefiting EV engine performance. Pure a-C lacks the conductivity needed for this crystallization effect, which can be improved by doping the a-C with metallic elements. Using copper or nickel as dopants can reduce electrical resistivity and catalyze carbon nanostructure formation, further reducing friction. This study investigates the tribological behavior of metal-doped a-C coatings under electrified ball-on-plane tests. Me:a-C coatings were deposited on glass substrates using pulsed DC balanced magnetron sputtering. Ni and Cu were used as dopants, with different concentrations, to improve electrical conductivity. Tribological tests involved a ball-on-plane setup with a 10 N normal load, 5 mm stroke, and 0.28 Hz frequency, applying 30 V in four current flow modes: current flowing from ball to plane, from plane to ball, no current, and intermittent on-off cycling every minute. The coatings' microstructure and composition were analyzed using Scanning Electron Microscopy with Energy-dispersive

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

2:40pm MC3-2-WeA-3 Graphene-Related Materials: Bridging Fundamental Tribology and Industrial Applications Across Multifarious Environments, Mingi Choi [ds602847@gmail.com], Ji-Woong Jang, Pusan National University, Republic of Korea; Anirudha Sumant, Argonne National Laboratory, USA, India; Ivan Vlassiouk, Oak Ridge National Laboratory, USA, Russian Federation; Jae-II Kim, Korea Institute of Materials Science, Republic of Korea; Young-Jun Jang, Korea Institute of Material Science, Republic of Korea; Songkil Kim, Pusan National University, Republic of Korea Solid lubricants play a crucial role as alternatives to liquid lubricants in extreme environments and as solutions for enhancing mechanical system performance under ambient conditions at the macroscale. Among these, graphene, a representative two-dimensional nanomaterial, has attracted significant attention due to its exceptional nanoscale tribological properties. However, its application as a solid lubricant for macroscale industrial systems remains a challenge. Recent studies have highlighted that tailoring graphene's properties through functionalization, oxidation, can significantly enhance its performance. This underscores the strong correlation between the tribological behavior of graphene-based materials and their elemental and compositional properties.

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

4:00pm MC3-2-WeA-7 Structure and Tribo-Mechanical Properties of Si-Containing ta–C Thin Films Grown by Cathodic Arc Evaporation, Nelson Filipe Lopes Dias [filipe.dias@tu-dortmund.de], TU Dortmund University, Germany; Domic Stangier, Oerlikon Balzers Coating Germany GmbH, Germany; Julia Urbanczyk, Gabriel Brune, Jörg Debus, Wolfgang Tillmann, TU Dortmund University, Germany

Among various types of diamond-like carbon, tetrahedral amorphous carbon (ta-C) thin films have attracted considerable attention due to their high hardness of up to 70 GPa, low friction, and high wear resistance. This property profile makes ta-C a particularly promising thin film system with broader application potential compared to the well-established hydrogenfree amorphous carbon (a-C) and hydrogenated amorphous carbon (a-C:H). For both a-C and a-C:H, thin film properties are typically tailored by incorporating modification elements to meet specific application requirements. In this context, silicon (Si) is widely used to improve the thermal stability and reduce friction under dry sliding conditions. As a result, the modification of ta-C by Si lies within the focus of recent research to tailor its thin film properties.

A key challenge in the synthesis of ta-C:Si is the precise incorporation of low Si concentrations into the thin film without significantly reducing the high fraction of sp³-coordinated carbon (C) bonds, which would compromise the superior hardness of ta-C. To overcome this, Si-containing graphite with 2.5 and 5 at.% Si as well as pure graphite were used as cathode materials and were mounted on an array of cathodic arc evaporators arranged vertically. By positioning AISI M2 steel substrates at different heights in front of the

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evaporators, Si-containing ta-C thin films with low Si concentrations were successfully deposited.

This vertical cathode arrangement allows for tailoring the Si content gradually decreases from top to bottom and for reaching a Si-free ta-C thin film at the lowest height. The hardness of ta-C:Si decreases with increasing Si content but remains above 40 GPa even at the highest Si concentrations. Notably, a high hardness exceeding 60 GPa is achieved at the lowest Si content. To correlate the mechanical properties with the structural characteristics, Raman spectroscopy with UV laser excitation was performed for precise structural analysis of the sp³-coordinated C bonds. Additionally, tribometer tests were conducted to evaluate the influence of Si content on the friction and wear behavior at room temperature. The results highlight the potential of depositing low Si-containing ta-C:Si thin films with superior tribo-mechanical properties using cathodic arc evaporation and low Si-containing graphite cathode materials.

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Tribology and Mechanics of Coatings and Surfaces Room Palm 3-4 - Session MC1-1-ThA

Friction, Wear, Lubrication Effects, & Modeling I Moderator: Michael Chandross, Sandia National Laboratories, USA

1:20pm MC1-1-ThA-1 Solid Lubrication in Thin Films: Mechanisms, Materials, and Performance, Daniel Pölzlberger, Institute of Materials Science and Technology, TU Wien, Austria; Rainer Hahn, Tomasz Wojcik, Philip Kutrowatz, Christian Doppler Laboratory for Surface Engineering of High-performance Components, TU Wien, Austria; Klaus Böbel, Julien Keraudy, Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein; Szilard Kolozsvári, Peter Polcik, Plansee Composite Materials GmbH, Germany; Philipp G. Grützmacher, Carsten Gachot, Institute of Design Engineering and Product Development, Research Unit Tribology, TU Wien, Austria; Helmut Riedl [helmut.riedl@tuwien.ac.at], TU Wien, Institute of Materials Science and Technology, Austria

Tribological contacts play an essential role in the prevalent and required endeavor for increased sustainability and efficient use of resources. Considering the energy losses related to friction and wear, a huge possibility of saving resources, energy, and CO2 is often overlooked. Here, solid lubricants are an attractive option, especially for applications pushing their conventional liquid counterparts to their thermal and chemical stability limits – typically at elevated temperatures above 200 °C or under extreme conditions excluding liquids (i.e. space industry, semiconductors, or life science). Therefore, this study examines different solid lubrication concepts in thin film materials, classifying them concerning predominant mechanisms, application ranges, and performance.

As a starting point, carbon-containing thin film materials will be discussed comprising diamond-like carbon (DLC) coatings and non-reactively sputter deposited transition metal (TM) carbide thin films (i.e., HfC, TaC, or WC). Here, advances in PVD growth techniques (i.e., HiPIMS) and their impact on tribological performance are in focus. Furthermore, insights on the limits of carbon as the source for solid lubrication will be given by a set of highresolution characterization techniques (i.e., HR-TEM, APT, etc.). The second part presents an alternative class of TM dichalcogenide coating materials (compared to MoS₂) and their in-situ formation. In detail, in an innovative approach, selenium nanopowders are converted in-situ into lubricious 2D selenides on sliding W and Mo films, achieving a coefficient of friction (COF) down to 0.1 in ambient air. This in-situ formation is an exciting concept, especially for extreme environmental conditions. Nevertheless, further advances in solid lubricants are required to overcome the limitations for high-temperature applications (above 450 °C). Here, a concept on B₂O₃ formation in TM borides (i.e., $TiB_{2\pm z}$ or $WB_{2\pm z}$) leads to a drastic reduction of COF from 0.6 to 0.2 at 500 °C (and higher temperatures), highlighting the capabilities of boron-containing thin films in high-temperature tribological contacts.

In summary, the different concepts of solid lubrication in thin film materials emphasize the potential of exploring new materials and the need for an indepth understanding to push these materials in potential applications.

2:00pm MC1-1-ThA-3 Study of Transparent Coatings for the Preservation of Colored Titanium Surfaces, Sarah Marion, Renée Charrière, Mines Saint-Etienne, France; Clotilde Minfray, Ecole Centrale de Lyon - LTDS, France; Laurent Dubost, HEF - IREIS, France; Jenny Faucheu, Mines Saint-Etienne, France; Vincent Fridrici [vincent.fridrici@ec-lyon.fr], Ecole Centrale de Lyon - LTDS, France

Although titanium is not a noble metal, it is increasingly attracting interest from the luxury industry (jewelry, watches, packaging) due to its lightweight, hypoallergenic properties, and especially the wide range of colors it can display when coated with a thin layer of TiO₂. However, its application in luxury products remains limited because these colors tend to lack durability. Improving the wear resistance of these colored TiO₂ layers, and in particular preserving the original color, is a critical challenge for luxury jewelry.

The interference-based nature of titanium's color makes it highly sensitive to changes in oxide layer thickness, as well as to variations in the oxide layer's chemical composition and internal structure, which can alter its refractive index. Tribological tests conducted on thin titanium oxide layers, using a100Cr6 steel ball in both dry conditions and with artificial sweat, demonstrated a clear correlation between color changes due to friction and a reduction in oxide layer thickness in both environments.

An experimental study of the wear resistance of several potential protective coatings deposited on oxidized titanium samples is carried out in order to preserve the color of the samples. Three coatings—SialON, Si $_3N_4$, and a commercial hydrophobic coating—were examined for their wear resistance in both dry and artificial sweat conditions, as well as for their transparency and surface wettability. The challenge is to have a coating that is not only transparent but also resistant to wear in both dry and sweat-exposed conditions and insensitive to fingerprints.

Thus, the color variation before and after coating, the surface wettability of the coating with water and sebum, as well as its resistance to dry friction and friction in the presence of artificial sweat against a 100Cr6 steel ball, will be analyzed and compared to those of uncoated TiO_2 to assess the performance of the coatings.

2:20pm MC1-1-ThA-4 Beyond Graphene: A ML-Assisted High-Throughput Molecular Dynamics Framework for Screening 2D Materials for Tribological Applications, Matteo Valderrama [m.valderrama23@imperial.ac.uk], Daniele Dini, James Ewen, Imperial College London, UK; Nicolas Fillot, INSA de Lyon, France

2D materials, with their unique atomic structures and tunable properties, have shown immense potential for achieving superlubricity (COF < 0.01) in sliding contacts. However, the vast design space of these materials presents a significant challenge in identifying optimal candidates for specific tribological applications. To date, only around ten 2D materials have been extensively studied for their tribological properties. This work explores a framework for applying machine learning (ML) assisted high-throughput molecular dynamics (MD) simulations to accelerate the discovery of highperformance 2D materials for tribological applications. 2D materials exhibit fundamentally different frictional behavior compared to their bulk counterparts, a phenomenon that can be observed at the atomic scale. To study this, our framework will computationally screen the tribological performance of thousands of 2D materials. By streamlining simulation cell generation and optimization, this framework facilitates the processing of tens of thousands of MD simulations. Combined with the recent advancements in GPU-powered simulations, this project could transform high-throughput MD, especially through the hybridization of both the computational approaches (CPU vs. GPU) and the implementation of interatomic potentials. The extracted tribological data will be used to train ML models, such as regressive random forests, LSTMS, and LLMs, to predict the performance of new materials. Our goal is to establish correlations between specific material properties and atomic friction mechanisms. gaining deeper insights into the underlying causes of atomic friction. We anticipate that this project will revolutionize the field of 2D materials by accelerating the design, prototyping, and experimental validation of materials that demonstrate robust superlubricity, making research more accessible and reproducible, and ultimately paving the way for their widespread adoption in various applications. In this presentation, I will delve into the details of our framework, demonstrate its validity, and present preliminary results on predicting friction in 2D materials based on their intrinsic properties.

2:40pm MC1-1-ThA-5 Nanoscale Wear of Metallic Multilayers - the Effect of Interface, *Tomas Polcar [polcar@fel.cvut.cz]*, *Ahmed AlMotasem*, Czech Technical University in Prague, Czech Republic

Extensive large molecular dynamics simulations (MD) were conducted to investigate the impact of different Zr/Nb interface orientations on the friction/wear behavior of Zr/Nb multilayers. The primary cause of plastic deformation of the Nb layer was dislocations and BCC twinning, while Zr layers deformed via dislocations and intrinsic stacking faults. The Zr/Nb exhibited better tribological properties, such as lower COF, higher scratch hardness, and improved wear resistance compared to their single-crystal counterparts. The interface structure was analyzed, and its blocking strength was discussed. tailoring them to achieve desired properties for specific applications.

The simulations of friction and wear were compared with experimentally obtained nanoscratches on Zr/Nb multilayers with a periodicity of 6 nm prepared by magnetron sputtering. The wear was evaluated by AFM, structure by STEM and XRD. Qualitative agreement with experiments demonstrates predictive power of MD simulations in tribology.

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3:00pm MC1-1-ThA-6 Electrification of Ti:MoS₂ Coatings for Tribological Applications, Newton K. Fukumasu [newton.fukumasu@gmail.com], Institute for Technological Research of Sao Paulo State, Brazil; Miguel R. Danelon, André P. Tschiptschin, Izabel F. Machado, Roberto M. Souza, University of São Paulo, Brazil

Next-generation adaptive coatings for heavy-loaded mechanical transmission systems enhance durability and efficiency by coupling external parameters, such as electrical conditions, with tribological performance, particularly relevant for electric vehicle powertrains and energy generation systems, where controlling friction and wear is crucial for improving operational efficiency. Also, in those systems, stray currents could be used for improving tribological aspects of mechanical systems. Coatings of transition metal dichalcogenides, such as molybdenum disulfide, promote excellent solid lubrication under high contact stresses and pure sliding conditions, but higher wear rates compromise coating durability. Metaldoping MoS₂ coatings allows the optimization of mechanical properties, including hardness and elastic modulus, promoting an amorphous coating structure and engineered coating bandgap. In this work, Ti:MoS2 coatings were deposited using a pulsed D.C. magnetron sputtering, with doping levels controlled by varying the power applied to Ti target. Tribological tests under electrified reciprocating conditions were conducted with uncoated AISI 52100 balls against Ti:MoS2 coated glass plates. Ti concentration was varied between 10 at% and 20 at% and electrified tests conditions considered positive, negative, and non-electrified contact with, when applied, a constant electric current of 100 mA. Ball movement frequency was set at 0.375 Hz with 4 mm stroke. Results indicated that friction was reduced under electrified conditions, particularly for coatings with lower Ti concentrations. Raman spectroscopy revealed recrystallized MoS₂ inside wear tracks, suggesting tribo-induced structural adaptation. Wider wear tracks and greater surface damage were observed when ball was positively charged. Results suggest that the electric field may promote differential migration of Mo, S, and Ti species, altering the tribofilm composition and morphology formed at the ball surface. This selective adsorption on the ball further enhances the formation of MoS2-rich regions, in which tribochemical reactions, enhanced by the electric current, may favor MoS₂ retention and regeneration at lower Ti concentrations, while higher Ti concentrations disrupt the lubricating behavior. The integration of tribology and electrification may lead to enhanced efficiency and durability of critical mechanical systems with selective surface chemistry and adaptive tribological performance.

3:20pm MC1-1-ThA-7 Wear Protection and Corrosion Resistance of Dlc Top-Layered Coatings with Nano-Multilayer Interlayer Structure, Adrián Claver, Institute for Advanced Materials and Mathematics (INAMAT2), Universidad Pública de Navarra (UPNA), Spain; Iván Fernández-Martínez, Nano4Energy, Spain; Pierre Collignon, Pd2i, France; Pablo Díaz-Rodríguez, Ambiörn Wennberg [Ambiorn.wennberg@nano4energy.eu], Nano4energy, Spain

Several industrial components are subjected to wear and corrosion, and they face significant challenges due to increased performance demands and the acidic and salty environments of industry. In this context, DLC coatings are presented as a great option to protect these components from corrosion as well as improving their performance by providing properties such as improved mechanical properties (higher hardness and wear resistance), chemical stability or low friction coefficient. In this study, different DLC top-layered coatings have been deposited by hybrid Physical Vapor Deposition and Plasma Enhanced Chemical Vapor Deposition (PVD-PECVD). The corrosion and wear resistance, as well as the mechanical properties and structure of coatings with different interlayer structures have been evaluated. In order to improve the protective properties of the coatings, different nano-multilayer structures based on TiNCrN, CrNOx or Cr/CrN have been deposited prior to the DLC top layer. In addition, the influence of the deposition parameters and the Si-addition to the DLC top layer have been studied and optimized. Chemical and structural properties of the coatings were evaluated by Raman spectroscopy, Glow Discharge Optical Emission Spectroscopy and scanning electron microscopy. Surface and mechanical characterization was performed by nano-indentation, confocal, Rockwell adhesion test and calotest. Wear resistance was evaluated using pin-on-disc tests with an applied load of 50 N, and wear tracks were analyzed by confocal. Finally, electrochemical and salt spray tests were used to study the corrosion resistance of the coating structures. Nano-multilayer structures showed improved wear and corrosion resistance, allowing the inhibition of localized corrosion in the DLC toplayer. Clear differences were observed in the DLC top layer due to the addition of Si. Although it would be necessary to test them in real

demanding working conditions, DLC coatings with nano-multilayers have shown to be a very interesting option to be used as an anti-corrosion and wear barrier coating.

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Tribology and Mechanics of Coatings and Surfaces Room Golden State Ballroom - Session MC-ThP

Tribology and Mechanics of Coatings and Surfaces Poster Session

MC-ThP-1 Role of Layer Position During Thermo-Mechanical Loading of Trilayers, Megan J. Cordill [megan.cordill@oeaw.ac.at], Claus O.W. Trost, Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Austria

Thermo-mechanical loading of thin films on rigid substrates is common method to assess film stresses as a function of temperature. However, these experiments have historically only been performed on single layer films even though multilayers are used in all advanced thin film technology. To illustrate the feasibility of measuring the thermo-mechanically induced stresses of multiple layers simultaneously, different architectures of brittle-ductile-brittle and ductile-brittle-ductile trilayers on silicon were heated with in-situ X-ray diffraction (XRD). The use of XRD provides individual film stress evolution simultaneously to understand delamination mechanisms of the trilayer architecture. The main aspects presented will be the strain evolution under thermo-mechanical loading as a function of layer position. Following Mo and Cu films from next to the substrate, to the middle position, and as the top surface film found that position in the trilayer architecture significantly influences the stress-temperature curve, thus the deformation mechanism due to thermo-mechanical loading.

MC-ThP-4 Nanoindentation and Micropillar Compression at Cryogenic Temperatures, Eric Hintsala [eric.hintsala@bruker.com], Kevin Schmalbach, Douglas Stauffer, Bruker Nano Surfaces, USA

Mechanical reliability at low temperatures is required for environments in energy and aerospace applications. Due to its highly localized measurement capabilities, nanomechanical approaches can be useful for isolating individual regions within a more complex microstructure or component or testing of thin films.In general, both modulus and yield strength gradually increase with decreasing temperature, but more sudden shifts in behavior can also be observed, such as phase transformations or ductile-to-brittle transitions. In situSEM testing enables visualization of the deformation mechanisms coupled with the measured mechanical properties helping complete the interpretation of the behavior. Alow temperature control system has been developed for the Hysitron PI89PicoIndenter (Bruker, USA) for in situ SEM testing that enables continuous temperature control from -130°C to 50°C. Independent temperature control on the tip and sample to enable proper temperature matching in vacuum and minimizes drift. The temperature dependent mechanical response of two metallic samples, Nitronic 50 and Tungsten, both by nanoindentation and micro-pillar compression.

MC-ThP-7 Investigating Arctic Environmental Effects on Dry Sliding Wear Behavior of Protective Coatings, Elyse Jensen [elyse.jensen@mines.sdsmt.edu], Austin McCracken, South Dakota School of Mines and Technology, USA; Emily Asenath-Smith, Cold Regions Research and Engineering Laboratory, USA; Grant Crawford, Forest Thompson, South Dakota School of Mines and Technology, USA

Understanding the tribological response of protective coatings to environmental conditions is required in order to tailor their functionality for extreme service conditions. This study establishes a methodology for evaluating the sliding wear performance of protective coatings in conditions representative of Arctic environments. A low temperature ballon-flat tribometer was modified to enable control over dewpoint within the testing enclosure. CrN-coated high strength stainless steel flats and alumina ball bearings were used as model wear couples. Dry sliding wear tests were performed on various CrN architectures at cold (-20 °C) and warm (30 °C) surface temperatures in low dew point air (<-20 °C). The repeatability of the testing approach was established by replicating environmental test conditions across multiple tests on the same flat sample. Wear scars were analyzed using laser scanning confocal microscopy and optical microscopy. Comparisons of coefficient of friction behavior as a function of sliding distance revealed that specific protective coating architectures respond differently to Arctic conditions.

MC-ThP-10 Validity of the 10% Rule of Thumb in Coatings Nanoindentation, Esteban Broitman [ebroitm@hotmail.com], EDB Engineering Consulting, France

When an indenter penetrates the surface of a film deposited onto a substrate, the mechanical response of the coating will be influenced by the mechanical properties of the substrate, according to its penetration depth h and the film thickness t. As the depth of penetration h increases, more of the mechanical contribution will come from the substrate.

The first who tried to separate the contribution of the substrate from the total measured hardness at the microscale was Bückle, who suggested a 10% rule of thumb: to indent no more than 1/10 of the film thickness to avoid the influence from the substrate. The rule has been adopted later by many researchers for nanoindentation experiments and extended also as valid for the elastic modulus. However, there are many experimental studies and numerical simulations showing that this rule is too strict for a hard coating on a very soft substrate and too loose for a soft coating on a hard substrate [1].

In this presentation, we will review the issue, and will discuss all factors that affect the maximum penetration depth for independent coating measurements. We will also present a simple experimental methodology that, in most of cases, gives the correct values for hardness and elastic modulus, independently of the coating/substrate system.

[1] E. Broitman, Indentation Hardness Measurements at Macro-, Micro-, and Nanoscale: A Critical Overview. Tribol. Lett. 65 (2017) 23.

MC-ThP-11 Enhancing the High-cycle Fatigue Strength of Ti-Al-N Coated Ti-6Al-4V by Residual Stress Design, Arno Gitschthaler [arno.gitschthaler@tuwien.ac.at], Rainer Hahn, Lukas Zauner, Tomasz Wojcik, TU Wien, Institute of Materials Science and Technology, Austria; Florian Fahrnberger, Herbert Hutter, TU Wien, Austria; Anton Davydok, Christina Krywka, Helmholtz Zentrum Hereon, Institute of Materials Physics, Germany; Jürgen Ramm, Anders Eriksson, Oerlikon Balzers, Oerlikon Surface Solution AG, Liechtenstein; Szilard Kolozsvari, Peter Polcik, Plansee Composite Materials, Germany; Helmut Riedl, TU Wien, Institute of Materials Science and Technology, Austria

Physical vapor deposited ceramic coatings are widely utilized to protect components operating in harsh environments, yet their influence on the high-cycle fatigue behavior of metallic substrates remains a subject of debate. In this study, the residual stress-dependent effect of arc evaporated TiAIN-based thin films on the fatigue life of Ti-6Al-4V was investigated. By employing various stress-modifying strategies — (i) including a substrate bias variation, (ii) a tantalum-based alloying approach, (iii) and a tailored interlayer design — we systematically modified the residual stress profiles within the coating and interface near substrate region. High-cycle fatigue tests, performed in a single cantilever configuration using a dynamic mechanical analyzer, revealed that a sufficiently pronounced residual compressive stress state within the TiAIN layer is critical to preventing premature failure. Once the residual compressive stress field effectively shifts fatigue crack nucleation into the bulk material, an improvement in the high-cycle fatigue limitof over 50% was achieved compared to the uncoated titanium alloy (from 420 MPa to 628 MPa at 10⁷ cycles).

To clarify the underlying mechanisms, a combination of high-resolution characterization techniques — namely high-resolution transmission electron microscopy (HR-TEM), transmission electron backscatter diffraction (t-EBSD), time-of-flight secondary ion mass spectrometry (ToF-SIMS), transmission X-ray nanodiffraction (CSnanoXRD), and micromechanical synchrotron-based experiments at DESY's PETRA-III — was employed. These experimental insights were integrated into a simple linear-elastic stress-failure model, providing an analytical framework to support the experimentally observed fatigue enhancements. The study not only resolves previous contradictory findings regarding the detrimental versus beneficial effects of hard ceramic coatings on fatigue performance but also establishes clear criteria for optimizing coating design. In particular, our results demonstrate that an optimized residual stress distribution is key to deploying the full potential of HCF-resistant TiAIN-based coatings. Adjusting process parameters and designing the interlayer helps maximize TiAIN coatings' effectiveness for extending the lifespan of Ti-6AI-4V parts.

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Tribology and Mechanics of Coatings and Surfaces Room Palm 3-4 - Session MC1-2-FrM

Friction, Wear, Lubrication Effects, & Modeling II

Moderators: Julien Keraudy, Oerlikon Balzers Coating AG, Liechtenstein, Pantcho Stoyanov, Concordia University, Canada

8:00am MC1-2-FrM-1 Linking Atomic-Scale Surface Structure and Friction via Multiscale Modelling: The Case of Carbon-Based Coatings and Tribofilms, Gianpietro Moras [gianpietro.moras@iwm.fraunhofer.de], Fraunhofer IWM, MicroTribology Center 2TC, Germany INVITED

Carbon surfaces play a fundamental role in tribology. There is not only the case of carbon-based coatings, but also the less obvious case of low-friction, carbon-based tribofilms deposited on other materials by liquid or solid lubricants. In all cases, friction in dry and boundary lubrication conditions depends on the atomic structure of the sliding surfaces. A stable chemical passivation of surface dangling bonds is a prerequisite for low friction and wear. However, even subtle changes in surface chemistry can cause the friction coefficient of passivated carbon interfaces to vary significantly. In this talk, I will present the results of multiscale simulation studies that combine quantum mechanics, molecular dynamics and contact mechanics to shed light on the relationships between the chemical structure of carbon surfaces and friction.

I will initially focus on superlubricity (friction coefficient < 0.01) with diamond-like carbon coatings and silicon nitride. Stable superlubricity over a wide range of operation conditions has been recently achieved at Fraunhofer IWM in plain-bearing test rigs using glycerol as a lubricant. Hydrodynamic superlubricity with glycerol is possible at high temperature and facilitated by the presence of water. However, the mechanisms responsible for superlubricity in boundary lubrication with glycerol are still under debate. Our simulations reveal a complex mechanochemical process involving the tribochemical decomposition of glycerol molecules at surface asperity contacts, the plastic deformation of the resulting H-, O- or N-containing amorphous carbon tribofilm and the formation of partially aromatic surface regions. These smooth and unreactive surfaces enable superlubricity even when asperity contacts run dry or are separated by nanometric, highly viscous glycerol films.

In the second part of my talk, I will extend the study to the effects of boron and fluorine. Our simulations suggest that hydroxyl groups that normally passivate carbon surfaces in humid environments can be activated by boron and form B–O dative bonds across the tribological interfaces, leading to a mild friction increase. Surface passivation by C–F bonds, instead, is very stable. This is the basis of the exceptional tribological properties of some perfluorinated carbon materials, but also of their accumulation in the environment and in biological systems. Our simulations provide answers to open questions about their friction mechanisms that may be useful in the search for alternatives: Why are perfluorinated carbon surfaces polar and hydrophobic? Why are they more slippery than their hydrogenated analogues? Why is PTFE non-sticky but forms transfer films on PTFE-lubricated steel surfaces?

8:40am MC1-2-FrM-3 Effects of Graphene Additives on the Mechanical Properties and Corrosion Resistance of Plasma Electrolytic Oxidation Coatings on AZ31B Magnesium Alloy, Guan Zhong Chen [M11188020@0365.mcut.edu.tw], Department of Materials Engineering, Ming Chi University of Technology, Taiwan., Taiwan; Chuan Ming Tseng, Center for Plasma and Thin Film Technologies, Ming Chi University of Technology, Taiwan., Taiwan

Graphene, as a two-dimensional layered material, exhibits excellent electrical conductivity and superior friction and wear resistance, making it widely applicable in various industries. In this study, the ceramic composite coatings on AZ31B magnesium alloy were prepared by using plasma electrolytic oxidation (PEO) in alkaline solutions with sodium phosphate, sodium silicate, potassium fluotitanate and graphene additions. The effect of graphene content on mechanical properties and corrosion resistance of PEO coatings on AZ31B magnesium alloy was investigated. The microstructural characteristics and compositional analysis of the PEO coatings were examined by using field emission scanning electron microscopy (FE-SEM), X-ray energy dispersive spectroscopy (EDS), and X-ray diffraction (XRD). The micro-Vickers hardness tester, nanoindentation instrument and a pin-on-disk tribometer were employed to measure the hardness and wear resistance of PEO coatings. The potentiodynamic polarization measurements and salt spray test were conducted to evaluate the corrosion behaviors of PEO coatings in NaCl containing circumstances. The experimental results revealed that as increasing the graphene content from 25 mg/L to 75 mg/L, the thickness of PEO coating increased from 24.7 μm to 37.5 μm and the porosity decreased from 13.03% to 7.82%. The results of XRD and SEM-EDS indicated that the PEO composite coatings were mainly composed of MgO and Mg2SiO4. The hardness of PEO coating was increased with increasing the graphene content and the optimal hardness 1696 HV attained on the PEO coating with 75 mg/L of graphene addition. The potentiodynamic polarization curves in a 3.5 wt% NaCl solution showed the corrosion current density decreased with increasing graphene addition and the highest polarization impedance achieved for the PEO coating with 75 mg/L of graphene addition. Furthermore, the results of salt spray test after 14 days-exposure indicated that the PEO coatings with graphene addition exhibited fewer and smaller corrosion pits as compared to the PEO coating without graphene addition. In summary, the mechanical properties and corrosion resistance of PEO coatings were pronouncedly improved by graphene nanosheets incorporation.

9:00am MC1-2-FrM-4 Tribology of Protective CrN Coatings in Arctic Environmental Conditions, Forest Thompson [forest.thompson@sdsmt.edu], Elyse Jensen, Nathan Madden, Grant Crawford, South Dakota School of Mines and Technology, USA

The friction and wear behavior of protective CrN coatings has been shown to be highly sensitive to Arctic environmental conditions, such as the combination of cold temperatures (<20 °C) with low dew points (<-30 °C). To advance the mechanistic understanding of the tribological response of CrN to Arctic environments, the relationships between coating architecture, environmental conditions, coefficient of friction, and wear resistance were investigated. A series of CrN coatings were deposited onto stainless steel substrates with varying adhesion layer compositions (Cr, Ti, CrN) by reactive pulsed DC magnetron sputtering. Microstructural characterization of the asdeposited coatings was conducted via laser scanning confocal microscopy, electron microscopy, energy dispersive x-ray spectroscopy, and x-ray diffraction. Linearly reciprocating sliding wear tests were conducted using a ball-on-flat tribometer. The tribometer was equipped with an active cooling stage and a dry air source to achieve coating surface temperatures and environmental dew points representative of conditions that would be encountered in Arctic service environments. After tribological testing, focused ion beam milling and transmission electron microscopy were utilized to analyze specific sites within wear scars and to characterize wear debris structure. The results from this work contribute to efforts related to the design of protective coatings for extreme environments, such as those encountered at Earth's polar regions.

9:20am MC1-2-FrM-5 Impact of Gaseous Environments on the Tribological Performance of Steel and Advantages of DLC Coatings, *Pierre-Francois Cardey [Pierre-Francois.Cardey@cetim.fr]*, Cetim, France INVITED

The tribological performance of materials is strongly influenced by the gaseous environment, where composition and pressure alter wear and friction mechanisms. In particular, the energy and transportation industries are paying increasing attention to hydrogen-related issues due to its potentially embrittling effects and impacts on tribological performance. At CETIM, a pin-on-disc tribometer was developed to analyze these interactions under various gaseous atmospheres across a wide range of temperatures and pressures.

This study focuses on two steel grades (high carbon and chromium steel 52100, and austenitic stainless steel 316L), tested in nitrogen, helium, and hydrogen atmospheres, with variations in contact pressure, temperature, and sliding speed. The results highlight how these environments affect the formation of protective oxide layers, which play a key role on friction and wear. The effects of hydrogen are also specifically studied due to its embrittling and reducing properties.

In this context, Diamond-Like Carbon (DLC) coatings emerge as a promising solution, acting both as a barrier to hydrogen diffusion and as a tribological enhancement in harsh gaseous environments. This study provides a comprehensive approach to optimizing material selection and surface treatments to improve the durability of components exposed to challenging industrial gaseous atmospheres.

10:00am MC1-2-FrM-7 Sliding Wear Behavior of Borided Ti₆Al₄v Alloy Under Dry Conditions and Simulated Body Fluids, J. A. Nieto-Sosa [antonio.nieto1094@gmail.com], G. A. Rodríguez-Castro, A. Meneses-Amador, INSTITUTO POLITECNICO NACIONAL, Mexico; E. E. Vera-Cárdenas, INSTITUTO TECNOLOGICO DE PACHUCA, Mexico; R. Pérez-Pasten-Borja, N. A. Hernández-Rosas, INSTITUTO POLITECNICO NACIONAL, Mexico

This study investigated the resistance to wear of borided Ti₆Al₄V alloy under dry conditions and in calf serum as simulated body fluid. The layers

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formation was conducted by powder-pack boriding at 1100 °C for 5 and 20 h of exposure time. The layer thicknesses less than 11 μm and hardness around 25 GPa were determined by optical microscopy and instrumented indentation, respectively. Through X -ray diffraction, TIB2 and TIB phases are identified, and the distribution of the chemical elements of B, Ti, Al and V are analyzed by energy-dispersive spectroscopy (EDS). Reciprocating sliding tests were performed with an ball of 6.35 mm of diameter as counterpart, setting a sliding distance of 100 m and loads of 10 and 20 N for both conditions. The tribological results show that the wear rate decreases in the hardened titanium. In addition, the effects of the SBF are studied on the friction coefficient and wear mechanisms.

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