Tribology and Mechanics of Coatings and Surfaces
Room Palm 1-2 - Session MC1-1-MoM

Friction, Wear, Lubrication Effects, and Modeling I

Moderators: Carsten Gachot, Vienna University of Technology, Austria, **Giovanni Ramirez**, Zeiss Industrial Quality Solutions, USA

10:00am **MC1-1-MoM-1 Modern Analytical Methods for Characterizing Wear Surfaces and Subsurfaces***, **Thomas Scharf (thomas.scharf@unt.edu)**,* The University of North Texas, USA **INVITED**

It is now common to employ focused ion beam (FIB)-SEM and subsequent TEM characterization techniques to study 'site-specific' deformation structures. In this talk, I will highlight more underutilized diffraction and imaging techniques, such as Precession Electron Diffraction (PED)-TEM, Transmission Kikuchi Diffraction (TKD)-SEM, as well as 3-D FIB serial sectioning to interrogate subsurface structural evolution during sliding wear. *First*, new insights into solid lubrication mechanisms in directed energy deposition (DED) metal matrix composites (Ni/TiC/graphite) reveal that the improved tribological behavior is due to the in-situ formation of a low interfacial shear strength amorphous carbon tribofilm that is extruded to the surface through refined Ni grain boundaries. 3-D FIB serial cross-sectioning inside the worn surfaces of these composites revealed that the tribological stresses in the subsurface extrude the graphitic, primary carbon towards the surface through intergranular separation of refined nanocrystalline Ni grains. *Second*, surface and subsurface structural evolution during sliding wear of an in situ nitrided DED titanium alloy, Ti-35Nb-7Zr-5Ta (TNZT), was studied by cross-sectional TEM coupled with PED. Corresponding precession–orientation imaging phase maps were used to determine the orientation and percentage of α and β-Ti in the worn nitrided TNZT. The maps revealed that the nanocrystalline grains of soft/compliant β are much smaller (10-100 nm) than hard/stiff α grains (>100 nm). Wear reduction is due to the combination of the above phases and increase in the alignment of {0002}-textured coarser α grains along the sliding direction with absence of texture in the highly refined β grains. *Lastly*, I will show how coupled cross-sectional TKD-SEM can interrogate the microstructural evolution in a Co-Cr alloy sliding on a Ta-W alloy.

10:40am **MC1-1-MoM-3 Wear-Protection Performance and Durability of *in-Situ*-Deposited Carbon TribofilmsDerived from Intrinsically Strained Cycloalkane Molecules as Lubricant Additives***, Z. Al Hassan, H. Wise, T. Martin, S. Liu, Q. Wang,* ***Y. W. Chung (ywchung@northwestern.edu)**,* Northwestern University, USA*; S. Berkebile,* US Army Research Laboratory, USA

Wear-protective coatings on tribo-component surfaces are usually applied *via* vapor deposition methods. Once worn, they can only be restored through component disassembly. In our study, we explored *in situ* carbon tribofilm deposition using intrinsically strained cycloalkane molecules. These molecules, when dissolved in lubricants, can induce tribopolymer formation under stress and temperature at asperities. Our previous work on cyclopropane-carboxylic acid (CPCa) as an additive in polyalphaolefin and dodecane demonstrated the successful deposition of micron-thick carbon tribofilms in 15 minutes during pin-on-disk testing with a ten-fold reduction in wear. New results show that even after the removal of CPCa from the lubricant, these tribofilms continue to provide wear protection for up to 40 hours. Detailed surface examination using Raman spectroscopy helps us unravel the underlying mechanism for such extended durability of these carbon tribofilms. This research suggests a unique approach to providing unlimited replenishment of wear-protective layers.

11:00am **MC1-1-MoM-4 Lubricant Interaction of Triboactive CrAlMoCuN Coatings in Steel Contacts***, K. Bobzin, C. Kalscheuer,* ***Max Philip Möbius (moebius@iot.rwth-aachen.de)**,* Surface Engineering Institute - RWTH Aachen University, Germany

Conventional lubricants are designed for wear and friction reduction in steel-to-steel contacts. Rising power densities require enhanced wear resistance of machine components. This can be achieved using hard CrAlN coatings, although their chemical inertness limits interaction with lubricants. Therefore, Mo and Cu are incorporated into CrAlN coatings, promoting tribo-chemical interaction with lubricants. Mo can interact with sulfur to create MoS_2 tribofilms. Cu acts catalytically for this reaction and can enhance tribofilm formation for Fe_x-P_y and a-C. In most applications like gear boxes, bearings or chain drives, it is economically and technologically challenging to coat all components. Therefore, this study focuses on coating-steel contacts. Three CrAlMoCuN coatings and one CrAlN reference were deposited using physical vapor deposition (PVD). Coating characterization includes morphology, coating thickness, chemical composition, indentation hardness, surface roughness and compound adhesion. All coatings, along with an uncoated reference, were tribologically investigated using a pin on disk (PoD) tribometer. As substrate, the chain pin steel 58CrV4 was used, quenched and tempered to H = (52±1,5) HRC. The PoD parameters were an initial contact pressure of p_{PoD} = 1,400 MPa, a relative velocity of v_{rel} = 0.1 m/s, and a temperature of T_{PoD} = 70 °C. 100Cr6 steel was used as counterpart and Polyalphaolefin (PAO) as lubricant. PAO was highly additivated with sulfur and phosphorous. Tribofilms were investigated using energy dispersive spectroscopy (EDS) and Raman spectroscopy. All CrAlMoCuN systems showed lower coefficients of friction compared to both references indicating the formation of MoS_2 containing tribofilms. This correlates with a significantly reduced total wear volume.Via EDS, Cu-enriched lubricant residues were found on the uncoated counterparts in the CrAlMoCuN system, indicating the interaction of Cu with the lubricant in the tribological contact for the first time. The results show high potential of CrAlMoCuN coatings for lubricated machine element applications.

Tribology and Mechanics of Coatings and Surfaces
Room Palm 1-2 - Session MC1-2-MoA

Friction, Wear, Lubrication Effects, and Modeling II

Moderators: Carsten Gachot, Vienna University of Technology, Austria, **Giovanni Ramirez**, Zeiss Industrial Quality Solutions, USA

1:40pm **MC1-2-MoA-1 Thermally Sprayed Hardmetal Coatings - Strategies for Replacement of WC-Co(Cr)***, **Lutz-Michael Berger (Lutz-Michael.Berger@ikts.fraunhofer.de)**, J. Pötschke, S. Conze,* Fraunhofer Institute for Ceramic Technologies and Systems IKTS, Germany **INVITED**

In 2023 the 100th anniversary of the invention of WC-Co was celebrated. It is one of the most successful composite materials ever and is applied as a sintered tool and wear protection, as well as a coating material. The reason for this success, are the extraordinary properties of WC, but also the nearly perfect interaction with Co as the binder metal. However, there are significant differences between sintered bodies and thermally sprayed WC-Co. These differences are the reason why nowadays WC-10Co4Cr is the standard material composition for highly wear-resistant coatings for service. As thermally sprayed coatings are serving more often at high temperatures and in corrosive environments, Cr_3C_2-NiCr is established as a second successful composite. The standard compositions have been developed on an empirical base, and have changed only little for decades, while there were significant changes in the feedstock powder production technologies.

However, there are currently several challenges which require to create alternatives with respect to environmental and health safety, supply, and of course technical performance. Reasons are the need to replace Co as the binder due to health and environmental issues but also the classification of both Co and W as critical raw materials.

Different possible strategies for partial (the binder only) and full WC-Co(Cr) replacement are currently investigated and will be discussed in detail. Thus, different complex binder alloys for WC-based thermal spray coatings are explored and compared with other alternative binder materials such as Ni or Fe-based alloys.

With respect to the full replacement of WC-Co(Cr) alternative hard materials are investigated:

1. Cr_3C_2 with additions of WC in order to improve the low-temperature wear properties of Cr_3C_2-NiCr
2. TiC/TiCN or NbC/NbCN as cubic hard materials with Ni- or Fe-based alloy binders
3. High-entropy carbides as the most recent development in this field.

One of the advantages of these alternative hard materials is their better compatibility with Co-free binder materials.

In order to develop effective coating material compositions, it is necessary to have a reliable approach for their selection. By using sintering bodies for this purpose, the interactions of the hard and the binder phase can be studied, and the potential of individual novel compositions can be evaluated.

2:20pm **MC1-2-MoA-3 Tribological Properties of Metallic Surfaces Obtained by 3D Additive Manufacturing (Laser Metal Deposition Process), for Repairing Applications***, T. ZURCHER, E. CHARKALUK,* Ecole Polytechnique, France*; **Vincent FRIDRICI (vincent.fridrici@ec-lyon.fr)**,* Ecole Centrale de Lyon - LTDS, France

The repair of worn metal parts represents a significant strategic challenge for industries. These repairs must be both economically and environmentally advantageous. Many ”conventional” repair processes are still used today to meet this need. However, most of these processes are not well-suited for performing fine repairs with complex geometries. An additive manufacturing (AM) process belonging to the Direct Energy Deposition family, called Laser Metal Deposition (LMD), addresses this specific need. This involves putting next to each other and stacking small weld joints called ”beads”, melting metal powder by projecting it under the focal point of a laser. The small diameter of the laser beam and the guidance of the nozzle’s movement by numerical control enable high-resolution repairs. The study of the mechanical properties of parts/repairs from this AM process has already been extensively addressed in the literature. However, very few studies have focused on their wear resistance property. This work thus focuses on the experimental study of the wear resistance of parts repaired by the LMD process, with the ultimate goal of providing methodological recommendations leading to repairs with good wear resistance. Inconel 718 and 316L stainless steel are studied in the case of a dry sliding flat-on-flat contact with reciprocating motion, under different conditions.

Through a detailed analysis of the wear of these repairs, a better understanding of their tribological behavior was acquired. Various wear modes based on tribological parameters, materials properties and LMD process parameters were highlighted. Furthermore, these studies have shown that the repair strategy and sliding direction do not significantly impact their wear resistance. Studies of residual stresses before and after wear tests have demonstrated that the inherent residual stresses of this process have a non-significant impact on wear. These studies have also shown that wear results are highly dependent on the repair material used and the tribological conditions applied on the repairs. Notably, it has been observed that 316L steel exhibits better wear resistance under similar tribological conditions. However, it has been demonstrated that IN718 repairs have a more competitive wear resistance compared to conventionally manufactured parts composed of the same alloy.

2:40pm **MC1-2-MoA-4 Improved Anti-Friction of Diamond-Like Carbon Incorporating Titanium***, **Jae-Il Kim (jaeil@kims.re.kr)**, Y. Jang, J. Kim,* Korea Institute of Materials Science (KIMS), Republic of Korea*; N. Umehara,* Nagoya University, Japan

Diamond-like carbon (DLC) is commonly introduced as a solid lubricant and anti-wear coating. On the other hand, the tribological performance of DLC is highly dependent on the intercontact with the mating material, which may result in high friction. The lubricity of DLCs is believed to be due to the carbonaceous transition layer formed on the mating materials. Industrially, steel- or copper-based alloys have been used as counterparts against DLCs to date, however, it is hard to achieve low friction in tribopairs with them due to the difficulty in forming a carbonaceous transfer layer.

We attempted to solve this problem by doping highly reactive titanium into DLC. Among many transition metals, titanium in particular has a large number of *d*-orbital vacancies, which can easily interact with the 2*p*-orbital of carbon. Therefore, we introduced the idea that this chemical property can promote the formation of a carbonaceous transfer layer, which we aimed to form a low-friction C/C contact interface to enhance the lubricity of the DLC/steel tribopair.

Ti-doped DLC was fabricated by co-depositing DLC with a filter cathode vacuum arc method and Ti with an unbalanced magnetron sputter. The tribological performance as a function of Ti concentration was investigated, and the tribofilms formed on the counterparts were chemically characterized. In conclusion, we report that the Ti-doped DLC exhibited enhanced long-term low-friction characteristic and superlubricity in various environments.

3:00pm **MC1-2-MoA-5 Tribological Study of Magnetron Spurrered W-S-(C) Thin Films Sliding Against Aluminium at High Temperatures***, **Todor Vuchkov (todor.vuchkov@ipn.pt)**, S. Jahan Sunny, A. Cavaleiro,* University of Coimbra, Portugal

Forming of Aluminium is often performed at elevated temperatures due to its poor formability and springback at room temperature. Forming of aluminium at elevated temperatures causes significant tribological issues like adhesive wear and galling, i.e. there can be significant material transfer from the workpiece to the tool/die. Due to the harsh conditions (temperatures up to 500°C), liquid lubricants cannot be utilized and solid lubricants can be an alternative. Self-lubricating thin films deposited by magnetron sputtering are good candidates for alleviating this issue, especially the ones containing transition metal dichalcogenides since they provide good lubrication in environments that lack humidity. In this study we deposited three types of films containing transition metal dichalcogenides (TMDs), of which one consisted only of WS_x and two films were alloyed with carbon (~27 and 35 at. % of carbon). We utilized various techniques for characterizing the physico-chemical properties of the deposited films like scanning electron microscopy, X-ray diffraction at elevated temperature and thermo-gravimetric analysis. The mechanical properties were assessed using scratch testing and nanoindenation. Tribological testing was performed against aluminium (1000 series) balls at room temperature, 200° and 400°C. The unalloyed WS coating had more porous columnar morphology while the carbon-alloyed ones showed increase compactness with reduced intercolumnar porosity. The thermal analysis indicated that the maximum operating temperature should be ~400-430 °C, for the pure WS coating, and a higher value of ~480-490°C for the carbon alloyed films. The thin films had good adherence to the tool steel substrates with an Lc2 critical load of 20-30 N and no gross delamination up to 70 N of load. The tribological results indicate that the unalloyed WS coating is the best solution for friction reduction against aluminium at the examined testing temperatures (up to 400°C). The carbon alloyed coatings also provided friction reduction but friction instabilities were observed and the film with the highest carbon content suffered excessive galling at 400°C. In terms of wear, the unalloyed WS coating generally suffered more wear compared to the other coatings. The results of the study present a high potential of the TMD-based sputtered coatings for applications involving sliding against aluminium at elevated temperatures.

4:00pm **MC1-2-MoA-8 Understanding the Tribological Mechanisms of TiO_2 Thin Layers: The Role of Composition and Structure of the Oxide Layer on Wear in Relation to Color Variation***, **Sarah Marion (sarah.marion@emse.fr)**, M. LENCI,* Mines Saint-Etienne, Université de Lyon, CNRS, France*; C. MINFRAY, V. FRIDRICI,* Laboratoire de Tribologie et Dynamique des Systèmes, Université de Lyon, Ecole Centrale de Lyon, France*; L. DUBOST,* IREIS, HEF group, France*; J. FAUCHEU, R. CHARRIERE,* Mines Saint-Etienne, Université de Lyon, CNRS, France

Although titanium is not a noble material, it is attracting more and more attention in the luxury industry (jewelry, watches, packaging) because of its lightness, hypoallergenic properties and, above all, the many colors it can produce when coated with a thin layer of TiO_2. However, its use in luxury goods is currently limited because its colors do not last very long. The wear resistance of thin colored layers of TiO_2, and especially the preservation of the original color, is an important issue for luxury jewelry.

The degradation of the color of anodized titanium is well studied in the field of architecture [1,2] and in this case, is mainly linked to a change of the thickness of the TiO_2 layer in the presence of acid rain. In addition, Diamanti et al [3] have shown that prolonged rubbing in an artificial sweat solution causes partial discoloration of the anodized titanium surface. However, the origin of the color degradation in this case has not been identified. The interferential origin of the color makes it particularly sensitive to a variation in thickness, but also to a variation in the chemical composition and internal structure of the oxide layer through a change of the refractive index.

The aim of this study is therefore to compare the wear resistance of thin TiO_2 layers produced by different processes. One is to anodize a Ti-6Al-4V substrate and the other is to deposit a TiO_2 layer by magnetron sputtering on a Ti-6Al-4V substrate. The influence of the chemical composition, crystallinity, internal morphology and thickness of the oxide layer on tribological behavior is studied and correlated with the change in color observed during dry rubbing and rubbing in the presence of artificial sweat.

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[2] M. Kaneko, M. Kimura, K. Tokuno, Corros. Sci. 52 (2010) 1889-1896.
[3] M. V. Diamanti, P. Pozzi, F. Randone, B. Del Curto, Materials and Design 90 (2016) 1085-1091.

4:20pm **MC1-2-MoA-9 Tribocorrosion Behaviours of TiZrNbTaFeC High Entropy Carbide Coatings by Superimposed HiPIMS and MF System***, **Ismail Rahmadtulloh (ismailrahmadtulloh2@gmail.com)**, C. Wang,* National Taiwan University of Science and Technology, Taiwan*; B. Lou,* Chang Gung University, Taiwan*; J. Lee,* Ming Chi University of Technology, Taiwan

Recently, the tribocorrosion issue has become crucial in various industry applications due to its impact on the failure and degradation of materials. In this study, TiZrNbTaFeC high entropy alloy carbide coatings (HEACCs) were deposited on the surface of AISI 52100 using a superimposed high power impulse magnetron sputtering (HiPIMS) and medium-frequency (MF) sputtering system. We observed an amorphous-like phase in coatings containing carbon within the range of 0 to 16.53 at.%. However, a transition to an FCC structure occurred when the carbon content increased to 32.4 and 35.8 at.%. The highest average hardness of 22.1 GPa was observed for HEAC#14 with a carbon content of 32.4 at.%. The coatings were immersed in a 0.5 H2SO4 solution at room temperature. At static corrosion, the carbon-free TiZrNbTaFe coating has a higher polarization ratio of 6.53×10^4 Ωcm^2 followed by HEAC#18 (35.8 at.% C) of 3.71×10^4 Ωcm^2, indicating excellent corrosion resistance. For the tribocorrosion tests, the coatings were subjected to 1 N load using a pin-on-disk tribometer at a sliding speed of 50 rpm. The wear rate and details of tribocorrosion studies on TiZrNbTaFeC high entropy alloy carbide coatings will be explored.

4:40pm **MC1-2-MoA-10 Friction and Wear of a-C:H and a-C:H:Si Coatings Sliding Against Different Counterpart Materials Under Dry and Moist Environments***, **Francisco A. Delfin (delfinf@frcu.utn.edu.ar)**,* National University of Technology, Regional Faculty of Concepción del Uruguay (UTN – FRCU), Argentina*; J. Jeoffrey,* Universiti Teknologi Petronas, Malaysia*; M. Schachinger, C. Forsich,* University of Applied Sciences Upper Austria*; S. Brühl,* National University of Technology, Regional Faculty of Concepción del Uruguay (UTN – FRCU), Argentina*; D. Heim,* University of Applied Sciences Upper Austria

The self-lubricating effect of DLC coatings is a very well-known feature, although they have yet to occupy a substantially influential position in mainstream tribological applications. This objective is increasingly critical due to the escalating worldwide focus on achieving energy efficiency, lowering fuel consumption and cutting environmentally harmful emissions. To reach these milestones, a deeper understanding of DLC coatings is required, namely regarding the intricate relationship of friction and wear rates within diverse tribosystems, where parameters such as relative humidity and the material of the counter body show decisive influence. In this work, DLC coatings were deposited using a modified commercially available PA-CVD system on AISI 4140 steel. Two kind of coatings were produced, a-C:H and a-C:H:Si, at temperatures of 450 °C and 550 °C. Process gas consisted of a mixture of argon, acetylene, and HMDSO as silicon precursor. Characterization was carried out by means of nanoindentation, Raman spectroscopy, as well as GDOES and EDX. Tribological behavior was evaluated by means of Pin-on-Disk, using the coated sample as the disk, a 12 N normal load, a speed of 0.4 m/s and a total sliding distance of 2000 m. Counterparts were 6 mm balls, of which three different materials were used: AISI 52100 bearing steel, Al_2O_3 and Si_3N_4. Test chamber was conditioned using forced air recirculation and beakers containing either water or regenerated silica gel to create a humid or a dry environment, respectively. Friction coefficient was registered during the entire test. The wear track was evaluated with optical and confocal microscopy, as well as SEM/EDX and Raman spectroscopy. Hardness and elastic modulus increased with deposition temperature, and the values were doubled with silicon doping. However, a lower friction coefficient and wear volume loss were found in Si-free samples. In general, the coatings showed varied responses to the different environments and counterparts: a-C:H showed oxidation with higher humidity, whereas a-C:H:Si exhibited high wear in the drier ambient, producing several peaks in the friction coefficient during the test. The steel counterpart exhibited a lubricious oxide layer that helped reduce the friction coefficient, thus performing better in the humid environment. The Si_3N_4 counterpart showed the highest adhesion when sliding against a-C:H:Si, although a rather low friction coefficient and wear was shown when testing the Si-free samples.

5:00pm **MC1-2-MoA-11 Evaluation of the Sliding Wear Performance of Binary CrN and Nanocomposite CrSiCN Coatings in Arctic Environments***, N. D'Attilio, **Forest Thompson (forest.thompson@sdsmt.edu)**, N. Madden,* South Dakota School of Mines and Technology, USA*; E. Asenath-Smith,* US Army Corps of Engineers Cold Regions Research and Engineering Laboratory, USA*; G. Crawford,* South Dakota School of Mines and Technology, USA

The efficiency, service lifetime, and durability of engineering components operating in the severe cold and dry environments found in Earth's polar regions can potentially be improved using protective coatings based on transition metal nitrides. However, the tribochemical wear behavior of these ceramic materials is particularly sensitive to operating conditions. Thus, there is a need to understand the influence of arctic environments on the sliding wear performance of these coatings. In this work, binary CrN, columnar CrSiCN, and glassy CrSiCN coatings were produced using filament-assisted reactive magnetron sputter deposition. The coatings were characterized using energy dispersive X-ray spectroscopy, X-ray diffraction, transmission electron microscopy, and atomic force microscopy. The mechanical properties of the coatings were tested using nanoindentation and the wettability of the coatings was determined using tilting-base contact angle goniometry. A ball-on-flat tribometer equipped with an active cooling stage and dry air source was used to assess the tribological performance of the coatings under various combinations of coating surface temperatures and environmental dewpoints. Ultimately, the microstructure and amorphous phase content was found to play a major role in the performance of CrN-based coatings in these environments. The wear resistance of all coating types was found to suffer under a combination of low surface temperature (-20 °C) and low dewpoint (-33 °C), while their frictional behavior under frosting conditions (-20 °C surface temperature, -

10 °C dewpoint) was primarily controlled by the presence of ice at the contact zone.

Tribology and Mechanics of Coatings and Surfaces
Room Town & Country B - Session MC2-1-TuA

Mechanical Properties and Adhesion I

Moderators: Jazmin Duarte, MPI für Eisenforschung GMBH, Germany, **Bo-Shiuan Li**, National Sun-Yat Sen University, Taiwan

1:40pm **MC2-1-TuA-1 Boosting Mechanical Properties of Metallic Thin Films Through Advanced Nanoengineered Design Strategies***, B. Francesco,* LSPM-CNRS, France*; A. Brognara,* Max-Planck-Institut für Eisenforschung, Germany*; P. Djemia, D. Faurie,* LSPM-CNRS, France*; A. Li Bassi,* Politecnico di Milano, Italy*; G. 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 **INVITED**

The current trend toward miniaturization in devices components in key technologies such as micro-/nanoelectronics, energy production, sensors and wear protection requires the development of high-performance nanostructured films with superior mechanical properties. Especially, mutually excluding structural properties such as high yield strength and ductility need to be combined, but also high adhesion with the substrate and large fatigue resistance. In order to trigger microstructure-induced material properties, control of the micro-scale structure, atomic composition, average grain size, and layer/film thickness must be optimized based on nanoengineering design concepts.

Here, I will present recent results for several class of advanced thin film materials including nanostructured metallic glasses (ZrCu/O, ZrCuAl/O...)[1-3] high entropy alloys (CoCuCrFeNi, Al/CoCuCrFeNi) and nanolaminates (fully amorphous, amorphous/crystalline)[4], showing how the control of micro-structure affect the and micro-scale mechanical behavior and enable ultimate mechanical properties.

Among the main results, I will show the potential of Pulsed Laser Deposition (PLD)[1, 2] as a novel technique to synthetize nanostructured cluster-assembled ZrCu, ZrCuAl/O, and CoCuCrFeNi films, reaching ultimate yield strength (>4 GPa) and ductility (>15 %) for ZrCuAl/O films. I will show how the control of the sublayer thickness (from 100 down to 5 nm) in fully amorphous nanolaminates influences the deformation behavior suppressing the shear bands formation, while tuning the mechanical properties with mutual combination of large ductility (> 10%) and yields strength (>2.5 GPa). Finally, I will show how alternating CrCoNi (crystalline)/TiZrNbHf (amorphous) nanolayers results in an high compressive yield strength (3.6 GPa) and large homogeneous deformation (~15%)[4].

Overall, our results pave the way to the development of nanostructured thin films with boosted mechanical properties and wide application range.

References:

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[2] C. Poltronieri et al., *Mechanical properties and thermal stability of ZrCuAlx thin film metallic glasses: Experiments and first-principle calculations*, Acta Mater. **258** (2023) 119226.
[3] A. Brognara, et al., *Effect of composition and nanostructure on the mechanical properties and thermal stability of Zr100-xCux thin film metallic glasses*, Mater. Design **219** (2022) 110752.
[4] G. Wu et al., *Symbiotic crystal-glass alloys via dynamic chemical partitioning*, Mater. Today **51** (2021) 6-14.

2:20pm **MC2-1-TuA-3 The Evolution of Residual Stress in the Immiscible Cr-W Alloy System***, **Tong Su (tong_su@brown.edu)**,* Brown University, USA*; J. Robinson, G. Thompson,* University of Alabama, USA*; E. Chason,* Brown University, USA

Metal alloy films are used in numerous technical applications such as magnetic storage, catalysis and hard coatings. As with any coating, residual stress is critical to their adhesion and physical properties. While there have been numerous studies of residual stress evolution in elemental metal systems under different processing conditions with corresponding mechanisms, there are far fewer in alloys.Such studies have been indispensable to understand adatoms evolution and residual stress development. In this work we study the stress in Cr-W which is a continuation of work performed in the Mo-V and W-V systems. These alloys are BCC elements that exhibit a miscibility gap which thermodynamically drive the elements to partition from the other. The measurements were performed at several deposition rates from 0.8 nm/s to 6.0 nm/s and pressures of 0.27 Pa and 0.47 Pa with the stress measured by an in-situ wafer curvature measurement technique. While thermodynamically driven to partition, the results indicate that alloy stress can be explained with similar mechanisms invoked for elemental systems. Nevertheless, the kinetic parameters that control the stress are a complicated superposition of the effects of the single elemental components.

2:40pm **MC2-1-TuA-4 Adhesion and Friction-wear Characterization of W-doped Hydrogenated Diamond-like Carbon (a-C:H) Coatings***, **Ihsan Efeoglu (iefeoglu@atauni.edu.tr)**, Y. Totik, G. Gulten, B. Yaylali, M. Yesilyurt,* Atatürk University, Turkey*; R. Gunay, G. Kara, B. Altintas,* TUSAS ENGINE INDUSTRIES (TEI), Turkey

AISI 4130 alloy steel, a significant material type for the majority of engineering applications in the industry, is used extensively in the aerospace, automotive, and defense industries due to its crucial characteristics, including high strength, durability, machinability, and corrosion resistance. Enhancing this material's surfaces mechanical and tribological characteristics with Tungsten (W)-doped diamond-like carbon (DLC) coatings stands out as a way to improve its performance in this investigation. Amorphous hydrogenated diamond-like carbon (a-C:H) coating has great mechanical and tribological properties. In this study, W-doped DLC coatings have been deposited on AISI 4130 via closed-field unbalanced magnetron sputtering. A L9 orthogonal array of the Taguchi method was utilized to optimize the variable coating parameters applied in the magnetron sputtering process. The microstructure, thickness, and composition of the a-C:H:W coatings were examined using scanning electron microscopy. The crystallographic characteristics of coatings were evaluated using X-ray diffraction in order to provide an extensive understanding of the coating structure. The specific measurements of the hardness and elasticities of the coatings were obtained using the nanoindentation test. The scratch test method was used to examine the adhesion properties of the coatings by determining their critical load values at which film delamination occurred. The tribological behavior of uncoated AISI 4130 substrate and a-C:H:W coating was determined with a pin-on-disc tribometer against an Al_2O_3 ball under dry sliding conditions. Delamination and gradual failures occurring in the wear test of the uncoated specimen increased the friction coefficient. On the contrary, the a-C:H:W coating exhibits such superior tribological properties that the friction coefficient decreased due to the prevention of delamination and gradual failures to a certain extent. It was observed that the scratch-adhesion properties of the coated specimens significantly contributed to the improvement of tribological performance.

3:00pm **MC2-1-TuA-5 Numerical and Experimental Evaluation of Cyclic Contact Loads on Titanium Borides***, A. MENESES AMADOR, G. RODRIGUEZ CASTRO, **Hugo Alberto Pérez Terán (hap_ter@hotmail.com)**, M. Melo-Pérez,* Instituto Politécnico Nacional, Mexico

In this work, an experimental and numerical study of the contact fatigue test on titanium boride (TiB_2/TiB) coatings is presented. The boride layers were formed at the surface of Ti6Al4V alloy using the powder-pack boriding process at temperatures of 1050 °C for10, 15 and 20 h of exposure times in order to obtain three different thicknesses. Optical microscopy was used to characterize the boride layer, the results showed an outer TiB_2 layer and an inner TiB layer (whiskers). The mechanical characterization (hardness and Young’s modulus) was carried out using Berkovich instrumented indentation technique. From the set of experimental conditions of the boriding process, cyclic contact loads were applied with a MTS Acumen equipment by repetitive impact of a sphere on the layer-substrate system. The experimental methodology consisted first determining the critical static load (load magnitude where cracks are observed) afterward cyclic sub-critical loads are applied with a frequency of 6 Hz for the three coatings. The test results indicate that the thinner coating exhibited better behavior under cyclic contact loads while thicker thickness showed greater damage under similar conditions. In order to evaluate the stress field generated in the boride layer during the application of static and dynamic loads, numerical simulations based in the finite element method were developed. The results showed good approximations with regard to contact diameters and residual depths obtained experimentally.

4:00pm **MC2-1-TuA-8 Mechanical Characterization of Nb-doped MoS_2 Coatings Deposited on H13 Tool Steel using Nb-based Interlayers**, ***Miguel Rubira Danelon (miguel.danelon@usp.br)**, N. Kyioshi Fukumasu,* University of São Paulo, Brazil*; A. Alves Carvalho,* Aeronautic Institute of Technology, Brazil*; R. Rodrigo Rego,* Aeronautics Institute of Technology, Brazil*; I. Fernanda Machado, R. Martins de Souza, A. Paulo Tschiptschin,* University of São Paulo, Brazil

Molybdenum disulfide is a 2D material with excellent lubricant properties, attributed to a low coefficient of friction (COF) resulting from weak Van der Waals forces between lattice layers and shear-induced crystal orientation. However, film oxidation harms its efficacy in humid atmospheres, leading to an increased COF and poor surface adhesion, making its use preferable in dry or vacuum conditions. To overcome these challenges, doping MoS_2 with elements, such as Nb, Ti, C, and N, emerges as a promising solution. Doping alters the reactions between the film and environmental oxygen atoms, reducing the environmental sensitivity of the film. Literature reports that doping MoS_2 with Nb promotes the formation of NbS_2 phases, which exhibit superior oxygen-trapping capabilities compared to conventional MoS_2 films, increasing its performance in humid conditions. Nevertheless, the adhesion of these coatings to a steel substrate present challenges and strategies involve the reduction of residual stresses and increase chemical affinity to the substrate by using of niobium-based materials as interlayer. Different interlayer characteristics, such as hardness, thickness and composition, can influence adhesion. A metallic niobium interlayer enhances film adhesion but, alternatively, a NbN interlayer, with ceramic characteristics, such as higher hardness, further improves adhesion. In this study, Nb-doped MoS_2 films were deposited on H13 steel using the pulsed direct current balanced magnetron sputtering technique. Different niobium-based interlayers were deposited to evaluate tribological behavior and adhesion properties of Nb-doped MoS_2 coatings. Scratch tests, conducted at room temperature and humidity, without lubrication, and under a progressive load, were performed to analyze the COF and adhesion of the coating, while instrumented indentation tests were conducted to assess the hardness and elastic modulus of the coatings. The Nb concentration of the films and interlayer was evaluated using scanning electron microscopy (SEM) with Energy-dispersive X-ray spectroscopy (EDS). Results indicated that a thicker interlayer optimally promoted the adhesion of the film, together with a high concentration of Nb on the interface between the Nb-doped MoS_2 film and the niobium-based interlayer. This result is justified by the increase of hardness led by higher Nb concentrations. Furthermore, better adhesion of the film promoted a low COF at greater load during tests. In summary, niobium-based materials can be used to enhance the adhesion properties of Nb-doped MoS_2 films and consequently improve their performance.

4:20pm **MC2-1-TuA-9 Mechanical Properties and Adhesion of Al Thin Films with Al_2O_3 Interlayers on Flexible Substrates**, ***Johanna Byloff (johanna.byloff@empa.ch)**,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland*; P. Renault,* University of Poitiers, France*; D. Faurie,* Université Paris-Saclay, France*; S. Husain,* University of Poitiers, France*; D. Casari, T. Edwards, B. Putz,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland

Metal thin films on polymers serve a variety of applications in the packaging, flexible electronics, and space sector. Challenges include mechanical failure during use, material restrictions, and non-recyclability. In the Aluminium (Al)-Polyimide (PI) system, favorable adhesive properties are attributed to an amorphous Al-O-C interlayer [1-3] (IL, 5 nm thick) between the metal film and the PI substrate. Through a combined atomic layer (ALD) and physical vapor deposition (PVD) setup, we are uniquely able to mimic interlayers artificially over a wide thickness range to study their mechanical and interfacial benefits. Using this setup, Al thin films (150 nm) with different Al_2O_3 interlayer thicknesses (0, 0.12, 1, 5, and 25 nm) were deposited on a polyimide substrate. These bi-layer samples were subjected to equi-biaxial tensile loading [4] with in-situ X-ray diffraction and electrical resistivity measurements at Synchrotron SOLEIL. The evolution of Al film stress, width of the Al diffraction peak and electrical resistivity could be determined as a function of IL thickness and applied strain. The Al and oxide layer thickness and microstructure as well as crack density and spacing after testing were investigated using scanning and transmission electron microcopy. Our results reveal a positive influence of the preceding ALD step on the mechanical properties of the Al thin films. All films with artificial ALD interlayers show reduced roughness and grain width in their PVD sputtered Al layers, resulting in a higher 0.2% yield stress, while overall maintaining ductile electro-mechanical behavior. This is possibly due to modifications of the PI substrate surface through ALD. Significant embrittlement was observed only in the Al film with 25 nm interlayer thickness. Most notably, comparison of a 5 nm artificial and natural interlayer shows a similar resistivity but a two-fold increase in yield strength in the artificial case. Adhesion between metal film and polymer substrate was evaluated with the tensile induced delamination method, indicating better adhesion (lower buckle density) with artificial Al_2O_3 interlayers.

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4:40pm **MC2-1-TuA-10 Buckling Structures, a Relevant Signature of the Mechanical Properties of Film/Substrate Systems**, ***Christophe COUPEAU (christophe.coupeau@univ-poitiers.fr)**,* Institut Pprime - CNRS - ENSMA - Université de Poitiers, France*; G. PARRY,* SIMAP, Grenoble-INP, CNRS, France*; J. DURINCK,* Institut Pprime - CNRS - ENSMA - Université de Poitiers, France

Thin films and coatings are used in a wide range of technological applications, such as microelectronics, packaging or optics. They often develop high residual stresses during the deposition process, sometimes about few GPa in compression. Such large compressive stresses may cause the nucleation and growth of buckling structures that generally result in the loss of functional properties that were initially conferred to such film/substrate composites. The aim of our studies is consequently to have a better understanding of the buckling phenomenon, by identifying the relevant parameters to prevent, to limit, or to control its occurrence.

From an experimental point of view, the fine investigation by optical and atomic force microscopies of the morphology of elementary buckling structures is consequently of great interest in order to qualitatively, or even quantitatively, extract some mechanical parameters of the film/substrate systems. Our studies concern for instance the effect of both elasticity [1-3] and plasticity [4] of the substrates on the maximum deflection of the buckles, the question of vacuum below the buckling structures [5], the effect of a pressure mismatch between the buckled film and its substrate on the occurrence of specific structures (such as donut-like or flower-like buckles) [6,7], the limit of the elastic theory framework to understand the buckling when plasticity is taking place in the film [8].

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Tribology and Mechanics of Coatings and Surfaces
Room Town & Country B - Session MC2-2-WeM

Mechanical Properties and Adhesion II

Moderators: Jazmin Duarte, MPI für Eisenforschung GMBH, Germany, **Bo-Shiuan Li**, National Sun-Yat Sen University, Taiwan

8:00am **MC2-2-WeM-1 *In Situ* Micromechanical Characterization of Thin Films: Strain Rate, Size and Microstructure Related Experiments in the SEM**, ***Szilvia Kalacska (szilvia.kalacska@cnrs.fr)***, CNRS LGF, Mines St. Etienne, France; *L. Petho,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland; *G. Kermouche,* Mines St. Etienne, France; *J. Michler,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland; *P. Ispanovity,* Eötvös Loránd University, Hungary **INVITED**

Creating multi-layered thin films with alternating dissimilar sublayers is proposing unusual (electric, thermal, optical, etc.) properties to be experimentally investigated. In such a system where grain size and texture can be controlled by the deposition/annealing process represents a unique opportunity to focus on some aspects of the deformation processes driven by the collective behaviour of dislocation. Our aim was to create a system with large enough grains (500-800 nm in diameter) and engineer flat grain boundaries to study plastic deformation modified by the presence of barriers.

A hybrid thin film deposition system (Swiss Cluster) was used to create the samples by combining atomic layer deposition (ALD) and physical vapour deposition (PVD) [1]. Sequential deposition of approx. 1 μm thick multilayers were separated by 10 nm thick Al_2O_3 interlayers. The initial 100-250 nm grain size was increased by extensive heat treatment (@800°C for 4h under Ar atmosphere, Fig. 1a). Such final specimen was quite challenging to create without porosities or major delamination from the substrate after heat treatment.

Afterwards, micropillars were fabricated using focused ion beam (FIB) milling close to the edge of the bulk sample (Fig.1b). These micropillars were then compressed at various strain rates (0.1-1000/s) using a nanodeformation setup (Alemnis AG). High (angular) resolution electron backscatter diffraction (HR-EBSD) was applied to study the geometrically necessary dislocation (GND) density distribution after low and high strain rate deformations. Sequential FIB-slicing [2] was applied to create 3D reconstructions of the deformed volumes.

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8:40am **MC2-2-WeM-3 Assessing Brittleness of Indium Tin Oxide Layers on Glass Substrates with Nanoindentation**, ***Kurt Johanns (kurt.johanns@kla.com)***, *S. Varma, J. Hay, B. Crawford,* KLA-Tencor, USA

Many of the materials used in manufacturing semiconductors are susceptible to cracking, i.e., exhibit brittle failure during processing and in application.While the definition of brittle is well understood, assigning a “brittleness” value to a given material or system of materials is not easy as “brittleness” is not a material property.Here, we define a simple set of nanoindentation experiments in an effort to assess the brittleness of Indium Tin Oxide (ITO) layers and provide feedback to semiconductor manufacturers looking to mitigate latent defects that may initiate and propagate during processing. Multiple ITO film thicknesses in different residual stress states of ITO are tested. Results show that indentation testing is capable of assessing the brittleness of ITO on glass when experimental and material system artifacts are taken into account. Multiple examples of nanoindentation and nanoscratch testing are provided with a focus on advantages and improved sensitivity over related techniques.

9:00am **MC2-2-WeM-4 The Effect of Nitrogen Flow Rate and Deposition Power on the Mechanical Properties and Microstructure of TiN Thin Film Deposited by HCD-IP Method**, ***Ching-Cheng Chen (moricechen@gmail.com)***, *K. Lan,* National Tsing Hua University, Taiwan

Due to the high strength-to-weight ratio, aluminum alloys are widely used in various industries. However, relatively low surface hardness limits the development of aluminum alloys. Various coatings deposited by PVD method have been proposed to enhance the surface hardness and corrosion resistance. Due to the excellent hardness, TiN coating is commonly used as a protective film. In addition, its dense structure effectively inhibits the corrosion of metal substrate. It was reported that substrate bias significantly affects the ZrN film structure deposited on AISI stainless steel 304, resulting in improved corrosion resistance [1]. Elevated deposition temperature helps the growth of ZrN thin film with a high quality. However, aluminum alloys might loss its hardness after high temperature depositions, and there are few articles discussing the effect of bias on the mechanical properties and corrosion resistance of TiN thin film when depositing under low-temperature by hallow cathode deposition ion-plating method (HCD-IP) on an aluminum alloy substrate.

Therefore, the purpose of this study was to investigate the effect of substrate bias on the mechanical and corrosion properties of TiN thin films coating on the aluminum alloy 6061. The TiN thin films were deposited by HCD-IP system under low temperature (<200°C). After deposition, the structure and texture of TiN thin films were confirmed by scanning electron microscope (SEM) and X-ray diffraction (XRD). Scratch test and nanoindentation were carried out to measure the adhesion strength and surface hardness. Salt spray test and polarization curve were used to evaluate the corrosion resistance of the TiN coated samples.

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9:20am **MC2-2-WeM-5 Effect of Metal Interlayers on Stress Relief of Mo_2N/Mo and Mo_2N/Ti Bilayer Coatings on Si Substrate by High Power Impulse Magnetron Sputtering**, ***Yun-Yang Sun (yysunk@gapp.nthu.edu.tw)***, *J. Huang,* National Tsing Hua University, Taiwan

The purpose of this study is to investigate the effect of different metal interlayers and interlayer thickness on the relief of residual stress in g-Mo_2N/metal bilayer coatings. Previous studies [1,2] have indicated that the metal interlayers such as Ti and Zr in transitional metal nitride/metal bilayer systems can significantly relieve stress, where the interlayer with a sufficient thickness can act as a buffer layer and relieve stress through plastic deformation, and the interlayer is usually under tensile stress state. However, when the thickness of interlayer is insufficient, the metal interlayer will act as a transitional layer to transfer the stress to the substrate. Mo is one of the metals that is commonly used in metal interlayer. However, from our previous study [1], the plastic properties of the metal interlayer, such as strength coefficient and strain hardening coefficient, may strongly affect the behavior of plastic deformation and change the capability of stress relief. Therefore, this study aimed to investigate the elastic and plastic properties of Mo and Ti interlayers on stress relief of Mo_2N coating on Si substrate. Mo_2N coatings were deposited on Si substrate by high power impulse magnetron sputtering (HiPIMS). The thickness of the coatings was controlled at about 1000 nm and Ti and Mo interlayers were set at 100, 150, 200, and 250 nm deposited by dc-UBMS. After deposition, the Mo/(Mo+N) ratio was determined by electron probe microanalysis, and the thickness of specimens was measured by the cross-sectional images from scanning electron microscopy and confirmed by the compositional depth profiles using Auger electron spectroscopy. X-ray diffraction were used to characterize the crystal structure and texture. The residual stress was measured by laser curvature measurement and average X-ray strain methods. Hardness and elastic constant were assessed by nanoindentation, and the atomic force microscopy was used to measure the surface roughness.

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9:40am **MC2-2-WeM-6 Microstructure and Mechanical Behavior of Magnetron Co-Sputtering Mo-Ta-N Coatings**, ***JIA-YI HSU (u0978750703@gmail.com)**, F. Wu,* Department of Materials Science and Engineering, National United University, Miaoli , Taiwan

The binary refractory metal nitride, Mo-Ta-N, coatings were fabricated and characterized in this study. The relationship between its microstructure and mechanical properties of the magnetron sputtering Mo-Ta-N coatings were investigated. The coatings were deposited using radio frequency reactive magnetron co-sputtering technique with input power control. The Mo-Ta-N thin films were prepared under a fixed inlet gas Ar/N_2 ratio and input power of 12/8 sccm/sccm and 150 W on Mo, respectively. The input power of Ta was tuned from 50 to 150 W to adjust the microstructure and composition. The addition amount of Ta and the deposition rate increased monotonically from 3.7 to 16.8 at.% and from 5.4 to 6.7 nm/min, respectively, as a function of Ta input power, while the (Mo+Ta)/N ratio kept a steady value around 1.0. The Mo-N film showed well-defined Mo_2N (111) and (200) facets with minor MoN (111) and (220) reflections. The Mo-Ta-N coatings exhibited a polycrystalline microstructure with MoN(111), Mo_2N(111), Mo_2N(200), TaN(111), TaN(200) and TaN(220) multiple phases and showed the nano-crystalline structure according to the broadened diffraction peaks. A maximum hardness of 18 GPa was found for the Mo-Ta-N coating deposited at an input power of 150/100 W/W. A sufficient adhesion was revealed and a better wear resistance was realized for Mo-Ta-N coatings with 6.8 and 10.4 at.% Ta and nanocrystalline multiple phase feature.

Keywords: refractory metal nitride, Mo-Ta-N, co-sputtering, multiple phase, nanocrystalline.

11:00am **MC2-2-WeM-10 Function of Mo Metal Interlayer in γ-Mo_2N/Mo Bilayer Coatings on D2 Steel Deposited by High Power Pulsed Magnetron Sputtering**, *Y. Fang, **Jia-Hong Huang (jhhuang@mx.nthu.edu.tw)**,* National Tsing Hua University, Taiwan

The purpose of this study was to investigate the function of Mo metal interlayer in the γ-Mo_2N/Mo bilayer coatings deposited by high power pulsed magnetron sputtering (HPPMS) on D2 steel substrate. The interlayer thickness was designed from 50 to 150 nm, and the thickness of γ-Mo_2N was controlled at 1000 nm. The results indicated no significant change in chemical compositions, microstructure, and mechanical properties of the Mo_2N coatings by adding a Mo interlayer. The residual stress of the bilayer coatings was measured by two methods. Laser curvature method was applied to measure the overall stress of the bilayer samples on Si substrate. The stress in individual layers was measured by the average X-ray strain method. For samples on Si substrate, the compressive stress in Mo interlayer was higher than that of the Mo_2N coating, which was also much higher than the yield strength of Mo ($\sigma_{y,Mo}$). This is quite different from the expected function that the interlayer can relieve stress by plastic deformation. The stress measurements on samples on D2 steel substrate showed that the Mo interlayer is under a very high compressive stress (> $6\sigma_{y,Mo}$), indicating that the interlayer cannot relieve stress by plastic deformation. Instead, the interlayer serves as a transitional layer that transfers the stress in Mo_2N to D2 steel substrate, where the D2 steel near the interface relieve the stress by plastic deformation, and the extent of stress relief is related to the interlayer thickness. The high strength coefficient and strain hardening exponent may be the reason that Mo cannot serve as a buffer layer but become a transitional layer. All samples show quite low wear rate, where the formation of the self-lubricating Magnéli oxides may be the major factor. Although the Mo interlayer can increase the adhesion strength of Mo_2N coating on D2 steel substrate, it is not necessary to add a Mo interlayer because the adhesion strength of Mo_2N on D2 steel substrate is sufficient for the wear test. Moreover, adding a Mo interlayer withinadequate thickness (50 nm) may not be beneficial to the stress relief of the Mo_2N coatings.

11:20am **MC2-2-WeM-11 Micro-Arc Oxidation of Commercially Pure Titanium Subjected to Hydrostatic Extrusion**, ***Lukasz Maj (l.maj@imim.pl)**,* Institute of Metallurgy and Materials Science, Polish Academy of Sciences, Poland*; F. Muhaffel,* Istanbul Technical University, Turkey*; A. Jarzebska, A. Trelka, D. Wojtas, K. Trembecka,* Institute of Metallurgy and Materials Science, Polish Academy of Sciences, Poland*; J. Kawalko,* AGH University of Science and Technology, Poland*; M. Kulczyk,* Unipress Extrusion, Poland*; M. Bieda,* Institute of Metallurgy and Materials Science, Polish Academy of Sciences, Poland*; H. Cimenoglu,* Istanbul Technical University, Turkey

Micro-arc oxidation (MAO) proved itself as very efficient method of the surface modification of so-called “valve metals” like titanium, aimed at improving their properties like wear resistance, bioactivity, antibacterial performance, etc. Enhancement of above-mentioned properties is connected with formation of well adhering oxide coating on the top of the substrate material thanks to the electrochemical reactions driven by the ions exchange between the substrate and electrolyte. Thus, not only the selection of proper electrolyte and MAO process conditions is important, but also microstructure of the substrate material. The plastic deformation of the substrate material may also affect the mechanisms of the oxide formation.Grain refinement and formation of higher density of low angle (LAGB) and high angle grain boundaries (HAGB) allows many more sites for the oxides nucleation to be formed what accelerates the coating formation. Application of severe plastic deformation techniques such as hydrostatic extrusion provided amazing results in terms of strengthening of metallic materials such as titanium, allowing for its widespread application, especially as future dental implants. Owing to the hydrostatic extrusion characteristics microstructure refinement down to the nanometric scale may be achieved, increasing strongly the density of LAGB and HAGBs, also having a huge impact on eventual surface modification with the methods like MAO.However, there is a lack of information about microstructure and tribological properties of the MAO coatings deposited on the surface of hydrostatically extruded titanium. In this work, titanium grade 4 substrates was subjected to 3-pass hydrostatic extrusion and subsequent rotary swaging reducing the initial diameter from 50 mm down to 5 mm.The rods were cut into diameter and subjected to MAO process in phosphate-based electrolyte with the help of the bipolar pulsed power supply.Such power supply allows for far better control of the electrochemical reactions during the deposition process than direct current or unipolar pulsed ones.Before the MAO coatings deposition, the cp-Ti substrates were thoroughly investigated in terms of determination of the HAGB and LAGB density with the SEM/EBSD method in order to determine their influence on the properties of the forming oxide surface layer. The microstructure observations (SEM/TEM) supported by phase and chemical analysis (XRD, SAED, EDS) allowed us discuss the mechanisms of oxide coating formation and correspond to their tribological behavior.

Acknowledgement: This research was funded by the National Science Centre of Poland, grant number UMO-2020/39/D/ST8/01783.

11:40am **MC2-2-WeM-12 Effect of Ultrasonic-Assisted Machining for Surface Functionalization of Innovative Work-Hardening Multi-Principal-Element Alloys**, ***Marcel Giese (marcel.giese@bam.de)**, D. Schroepfer, M. Rhode,* Bundesanstalt für Materialforschung und -prüfung, Germany*; B. Preuss, T. Lindner, N. Hanisch, T. Lampke,* Institute of Materials Science and Engineering (IWW), Chemnitz University of Technology, Germany

Multi-principal-element alloys (MPEAs) are an alloying concept consisting of at least two main alloying elements resulting in unique microstructures and potentially superior physical, mechanical and chemical properties, for instance a high work hardening capacity. These characteristics are determined by four core effects: sluggish diffusion, severe lattice distortion, high-entropy and cocktail effect. The development of MPEAs is a promising approach to extend the range of applications of conventional alloys by exploiting these core effects. In the present study, as reference to the conventional high-manganese steel X120Mn12 (ASTM A128), characterized by particularly high work hardening capacity generating exceptional mechanical properties, work-hardening MPEAs based on the equimolar composition CoFeNi in combination with Mn and C were developed. Specimens were produced as bulk material by melting via an electric arc furnace. In a second step the specimens undergo a surface finishing via milling process. Therefore, a hybrid milling process was used which, in addition to producing defined surfaces, also has the potential to reduce tool wear and increase surface integrity by introducing compressive stresses and increasing hardness through pronounced work hardening in comparison to conventional machining. The so-called ultrasonic-assisted milling (USAM) is characterized by an axial oscillation of the tool during the milling process. The machining parameters were varied to analyze the effect on work hardening together with process forces during milling and resulting surface integrity. Subsequently, microstructure evolution, hardness as well as resulting wear resisting capacity were investigated and correlated with the composition and the USAM parameters. For the MPEA CoFeNi-Mn12C1.2 a pronounced lattice strain and grain refinement due to the plastic deformation during the USAM was recorded, especially at high USAM amplitude and lower cutting speed due to the greater number of tool oscillations per cutting engagement. Consequently, a hardness increase of up to 380 HV0.025 was induced for the aforementioned MPEA exhibiting a higher wear resistance compared to the X120Mn12. This shows the promising approach for the development of work-hardening materials based on new alloy concepts such as MPEAs allowing also coatings required for applications in tribological systems. As conventional hard and wear-resistant coatings are challenging in machining due to massive tool wear this approach of functional coating materials with high hardening capacity during USAM have the potential to reduce tool wear and ensure a adequate surface integrity and wear resistance.

12:00pm **MC2-2-WeM-13 Metal/Oxide Nanolaminates of Al/Al2O3 by PVD-ALD: Understanding & Maximising Strength-Ductility**, ***Thomas Edwards (thomas.edwards@empa.ch)***, NIMS (National Institute for Materials Science), Japan*; B. Putz, T. Xie, L. Vogl, H. Jansen, A. Groetsch, M. Watroba, J. Michler,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland

The extent of the embrittlement in ductile-brittle multilayers often depends on the modulation period ($t_{brittle}$ + $t_{ductile}$) as well as on the modulation ratio ($t_{brittle}$/$t_{ductile}$) [1]. In this work, ductile-brittle multilayers of Al / Al_2O_3 / Al... were produced on Si substrates by a unique combination of atomic layer (ALD, Al_2O_3) and physical vapour deposition (PVD, Al) within a single deposition system. Using this ALD/PVD combination, neighbouring layer thicknesses can easily differ by one order of magnitude or more. In particular, the ability to deposit continuous sub-nm layers with ALD opens up a wide range of otherwise unachievable modulation and thickness ratios. The thicknesses and structures of the ALD layers were verified by HR-TEM imaging of lift-outs. The amorphous oxide layer thickness was previously optimised in the 0.1 nm – 10 nm range by microcompression, considering crack onset and propagation as a function of oxide layer thickness in tensile tested multilayer films. Here, the crystalline metal layer thickness is varied (10 nm – 250 nm) to optimise strength. The multilayer structure has good adhesion between individual layers and the oxide layers show increasing stretchability with decreasing film thickness. *In situ* TEM tensile loading was performed to evaluate the role of the amorphous-crystalline interfaces on dislocation motion in metallic layers, whilst microcompression at variable temperature and strain rate was used to quantify the activation parameters; this is compared with molecular dynamics simulations. The thermal stability of such multilayer films was also studied up to 0.9 T/T_m.

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Tribology and Mechanics of Coatings and Surfaces
Room Town & Country B - Session MC3-1-WeA

Tribology of Coatings and Surfaces for Industrial Applications I

Moderators: Nazlim Bagcivan, Schaeffler Technologies GmbH & Co. KG, Germany, **Stephan Tremmel**, University of Bayreuth, Germany, **Martin Welters**, KCS Europe GmbH, Germany

2:00pm **MC3-1-WeA-1 Tribological Coatings to meet Future Requirements for Green Mobility**, ***Steffen Hoppe (steffen.hoppe@tenneco.com)***, Tenneco Powertrain, Product & Technology, Germany **INVITED**

The global transportation industry is taking on the challenge of decarbonizing propulsion with the goal of achieving climate-neutral mobility. Hydrogen-powered internal combustion engines (H_2-ICEs) are the mix of applicable and complementary technology solutions. This technology can drive decarbonization on a broad scale, especially in commercial truck, off-highway and industrial applications. Non-fossil fuels like ammonia, methanol or e-fuels are required to achieve net-zero-CO2 emissions in marine and aviation markets.

The introduction of non-fossil fuels has a significant impact on the tribology systems in ICEs. Due to the significant differences in physical and chemical properties of hydrogen, ammonia, and methanol compared to gasoline and diesel fuel, the combustion strategies need to be adapted to these fuels. Advanced coating solutions are required for ICEs components to address the impact on the tribological systems caused by higher combustion temperatures, oil dilution or deterioration, and water entrapment.

This paper will show how the critical tribological system of piston rings can be optimized by developing high performance coatings. Hydrogen free DLC coatings, advanced thermal spray coatings and new electrochemical coatings are applied to achieve robust tribological systems in decarbonized propulsion systems.

2:40pm **MC3-1-WeA-3 Current-Induced Friction and Graphitization Effects in Amorphous and Tetrahedral Amorphous Carbon Coatings on M2 Steel: An Electro-Tribological Investigation**, *A. Khodadadi Behtash,* ***Ahmet T. Alpas (aalpas@uwindsor.ca)***, University of Windsor, Canada

In electric vehicles, protecting bearings from shaft voltages and bearing currents is key to avoiding premature wear and failure. Diamond-like carbon (DLC) coatings, with their low friction and insulating properties, could extend bearing life and reliability. This study assesses how electrical current affects the frictional behaviour of M2 steel coated with non-hydrogenated diamond-like carbon (a-C) and tetrahedral amorphous carbon (ta-C), by comparing their coefficients of friction (COF) against an AISI 52100 steel counterface under varying currents but the same loading conditions using a ball-on-disk tribometer. The uncoated M2 steel exhibited COF values ranging from 0.55 to 0.62, suggesting frictional instability and a tendency towards oxidation with sliding under electrical currents (Figure 1). The a-C coatings maintained a stable coefficient of friction under 0.15 up to currents of 1200 mA. In contrast, the ta-C coatings showed variable COFs, starting at 0.20 and rising above 0.60, indicating less stability under electrical currents. Micro-Raman analyses revealed graphene formation within the wear tracks of a-C samples upon exposure to induced current (Figure 2a). This current-induced graphitization within the wear tracks correlates with the a-C coating's low and stable COF. In contrast, ta-C coatings, with a higher sp^3 content, underwent less graphitization and more oxidation (Figure 2b) at the steel interface when subjected to the same electrical current. The increase in D peak intensity within wear tracks of a-C samples at higher currents suggested a rise in defect density in graphene layers formed. The mechanisms underlying these observations, including the interplay between graphitization and electrical current, as well as their implications for electro-tribological systems, will be discussed in the presentation.

3:00pm **MC3-1-WeA-4 Compositionally Graded MoS_2-WC Spray Coatings for Robust Tribological Protection in Low Viscosity Fuels**, ***Euan Cairns (euancairns@my.unt.edu)***, *J. Decker,* University of North Texas, USA*; S. Dixit,* Plasma Technology Inc., USA*; S. Berkebile,* Army Research Laboratory, USA*; D. Berman, S. Aouadi, A. Voevodin,* University of North Texas, USA

Increased usage of low carbon emission fuels, such as ethanol and dodecane, are driving a critical need for advanced lubricious materials to extend the wear life of fuel pump components. Solid lubricants are traditionally employed in applications where liquid lubrication is insufficient if not impossible. We demonstrated that molybdenum disulfide (MoS_2) and tungsten disulfide (WS_2) films spray-coated onto 52100 steel and WC-17Co surfaces decreased friction and wear during sliding in hydrocarbon fuels. Solid lubricant coatings were substantially more robust while sliding in non-polar dodecane fuel, where friction coefficients of less than 0.1 were maintained for thousands of sliding cycles. Meanwhile, in polar ethanol fuel, low friction was only kept for a few hundred cycles before sharp failure of the coatings, due to oxidation and removal of the lubricant from the wear track.

In this study, we propose to further enhance the wear resistance of crucial components in fuel pumps via designing compositionally graded MoS_2-WC coatings. The microstructure and wear data were evaluated using scanning electron microscopy (SEM) equipped with an energy dispersive spectrometer (EDS). Analysis of worn surfaces was performed using optical profilometry and Raman spectroscopy to analyze the chemical evolution inside the wear track across multiple fuel chemistries. Insights gained from this study offer valuable information for the development of robust lubrication solutions in the realm of low carbon emission fuel applications.

3:20pm **MC3-1-WeA-5 Tribological Behavior of DLC Coatings: Wear Map of Oil Lubricated Contacts in a Three-Pins-on-Disc Test Configuration**, *J. Keraudy, N. Manninen, F. Rovere,* ***Klaus Boebel (Klaus.Boebel@oerlikon.com)***, Oerlikon Surface Solutions AG, Liechtenstein

Diamond-like carbon (DLC) coatings have emerged as a promising coating solution able to combine high wear resistance and low friction coefficient. In fact DLC coatings comprise a family of different carbon based coatings which can show a broad range of properties based on the fraction of sp3/sp2 bonds and also on the amount of incorporated hydrogen or metal dopants.

In the present study different DLC coating variants were tested regarding their tribological performance.The coatings were tested in three pin-on-disc configuration under additive oil (ZDDP) lubricated conditions. Different pressure x velocity (P.V) conditions were tested during endurance tests in order to identify the coatings performance over a broad range of P.V conditions. The lubrication regimes were identified by Stribeck curves in order to determine the lubrication regimes for the different test parameters. The coatings were analyzed by scanning electron microscopy (SEM), profilometry and optical microscopy after the tribological tests, in order to evaluate the wear mechanisms. The coatings were characterized regarding their topography and morphology (by means of SEM analysis), roughness (by profilometry analysis) and hardness (by nanoindentation). The coatings chemical properties, roughness and hardness are strongly correlated with the tribological performance.

3:40pm **MC3-1-WeA-6 Structural and Tribo-mechanical Properties of AlCrVYON Thin Films with Varying O Contents Sputtered from Either AlCrVY or AlCrY and V Targets**, *W. Tillmann,* ***Finn Ontrup (finn.ontrup@tu-dortmund.de)***, *D. Aubry,* TU Dortmund University, Germany*; E. Schneider, M. Paulus, C. Sternemann,* Fakultät Physik/DELTA, TU Dortmund University, Germany*; N. Lopes Dias,* TU Dortmund University, Germany

The incorporation of Y and V into AlCrN has previously proven effective in enhancing oxidation resistance and tribological properties at elevated temperatures. As this improvement stems from the oxide formation of these elements, depositing O-containing AlCrVYN presents a promising approach for directly synthesizing thin films with enhanced tribo-mechanical properties for high-temperature applications. Therefore, AlCrVYON with varying O contents were deposited using a hybrid dcMS/HiPIMS process in two distinct approaches. In a reactive process, AlCrVYON was either sputtered from two AlCrVY targets or co-sputtered from AlCrY and V targets. For both configurations, the O_2 flow rate was varied from 0 to 20 sccm.

Sputtering from AlCrY+V targets results in higher O contents from 1.4 to 31.3 at.–% compared to the other target setup which achieves up to 20.7 at.–%. High-resolution x-ray diffraction using synchrotron radiation reveals a cubic CrN phase for all thin films, independent of the O content and target configuration. Nanoindentation tests show that the hardness stays at a high level above 40 GPa for an O content of up to 6.5 at.–% for AlCrVYON sputtered from AlCrVY targets and up to 14.7 at.–% for those sputtered from AlCrY+V targets. However, for higher O concentrations the hardness for both AlCrVYON variants decreases. Due to a constant decrease in the elastic modulus with an increasing amount of O, a maximum in the H/E ratio is observed for the aforementioned O contents. Furthermore, the tribological properties were analyzed using a high-temperature tribometer. No significant reduction of the coefficient of friction is noted at room temperature and only a slight improvement is visible in the higher temperature range for all AlCrVYON thin films, except the ones with the maximum *H*/*E* ratio. Annealing the thin films deposited from two AlCrVY targets for 2 h at 500, 600 and 700 °C demonstrates a high oxidation resistance for all AlCrVYON of at least 6.5 at.-% O, as no decrease in hardness, nor an increase in O content could be identified post-annealing. However, the thin films sputtered from AlCrY+V targets perform different in this regard, as the hardness decreases for all thin films after annealing at 600 °C.

In summary, the AlCrY+V target configuration produces AlCrVYON with higher O contents, resulting in a significantly different oxidation resistance. Other than that, both configurations show similar trends, demonstrating the advantage of adding small amounts of O into AlCrVYN. Thus resulting in a maximum of the *H*/*E* ratio for explicit O contents, depending on the target configuration.

4:00pm **MC3-1-WeA-7 Development and Process Optimization of Suspension Plasma Spray Coating to Enhance the Frictional Properties and Wear Resistance***, **Yong-Jin Kang (free83@kims.re.kr)**, Y. Yoo, S. Lee, D. Kim,* Korea Institute of Materials Science, Republic of Korea

Chromium oxide (Cr_2O_3) coating produced by the atmospheric plasma spray (APS) process is widely used in industrial fields such as anilox rolls, doctor blades in the paper-making industry, pump sleeves, and break discs that require resistance to sliding wear and corrosion. However, due to its low mechanical properties and high surface roughness and porosity, the development of alternative coating processes such as suspension plasma processes(SPS) is required. Accordingly, in this study, we developed a chromium oxide coating with low surface roughness, high hardness, and excellent wear resistance through a suspension plasma spray (SPS) process. By optimizing the stand-off distance, feedstock powder size, and power during SPS process, dense Cr_2O_3 coating with a porosity less than 2% was achievable. Microstructures and mechanical properties of as coated samples were characterized by SEM, XRD, surface profiler. Then, tribological properties, such as friction coefficient and wear rate, were evaluated by ball on disk test. The wear resistance of Cr_2O_3 coatings via SPS and APS processes was compared with their intrinsic microstructure and mechanical properties.

4:20pm **MC3-1-WeA-8 Excellent Tribological and Anticorrosive Properties of Nanocomposite Coating Based on Polyvinyl Alcohol/NiFe LDH/Tannic Acid***, **Dieter Rahmadiawan (n18127046@gs.ncku.edu.tw)**,* National Cheng Kung University (NCKU), Taiwan, Indonesia*; S. Chen Shi,* National Cheng Kung University (NCKU), Taiwan

This study investigates the effects of incorporating tannic acid into a polyvinyl alcohol (PVA)/MXene film. The composite was characterized for its mechanical, corrosion resistance, and tribological properties. The addition of tannic acid was found to enhance the mechanical strength of the composite, attributed to its crosslinking capabilities and interactions with the MXene nanosheets. Corrosion resistance was significantly improved, as tannic acid acted as a corrosion inhibitor, forming a protective layer on the composite surface. Tribological tests revealed reduced wear rates and improved frictional behavior, indicating the effectiveness of tannic acid in enhancing the lubricating properties of the PVA/MXene system. The comprehensive analysis presented in this study underscores the potential of PVA/MXene/Tannic Acid composites for applications demanding superior mechanical performance, corrosion resistance, and tribological efficiency.

4:40pm **MC3-1-WeA-9 Effects of Various Al/Cr Composition and Deposition Conditions on Surface Properties, Mechanical and Tribological Properties of AlCrN Coatings***, **SHINICHI TANIFUJI (tanifuji.shinichi@kobelco.com)**, M. NAKAMURA, R. TAKEI, S. KUJIME, T. TAKAHASHI,* Kobe Steel, Ltd., Japan

The environment in which tools and molds are used in production is becoming harder and harder year by year. Therefore, coatings applied by physical vapor deposition are required to have high hardness and oxidation resistance in order to extend the lifetime of tools and dies. Typical examples of coatings with such properties are AlTiN and AlCrN coatings. The oxidation resistance of coatings is attributed to the Al content of the coating, and it is known that the higher the Al content, the higher the oxidation resistance. On the other hand, the hardness of the coatings differs, with AlTiN and AlCrN coatings showing maximum hardness at 67 at% and 77 at% Al content, respectively. It is known that when the Al content in both coatings exceeds these levels, a decrease in hardness occurs due to the precipitation of soft AlN in the coating.

At last year's conference, KOBELCO introduced its new Cathodic arc evaporation system, AIP-iX. Using the new μ-Arc evaporation source installed in the system, KOBELCO reported on the surface properties of AlCrN coating with 75% Al content as observed by scanning electron microscopy, the coating hardness as measured by nanoindentation test, and the crystal structure of the coating by X-ray diffraction method. The results of the crystal structure of the coatings were also reported by the X-ray diffraction method. The results of the nanoindentation test and the crystal structure of the coating by X-ray diffraction are also reported. The surface smoothness of the coating is superior to that of the conventional cathodic arc deposition coating, and the cubic crystal structure is confirmed, indicating that a hard coating can be formed.

On the other hand, one of the main characteristics of μ-Arc is that it has fewer surface macro-particles than conventional coatings, but the effect of the surface properties of the coating on the tribological properties of the coating has not yet been clarified. In order to meet the needs of industrial applications, it is important to clarify the relationship between tool and die performance and the properties of the coatings. In this report, we describe the results of our studies of AlCrN coatings prepared under various compositions and deposition conditions, and the effects of these conditions on surface properties, mechanical and tribological properties, as well as the relationship of these coatings to tool and die performance.

5:00pm **MC3-1-WeA-10 Effect of Multilayer Architecture on Mechanical Properties and Cutting Performance of AlTiBN/AlCrBN Coatings***, **Chung-En Chang (abcd0214milk@gmail.com)**, Y. Chang,* National Formosa University, Taiwan

Ti-6Al-4V alloy is a currently popular material known for its high fracture toughness and hardness, making it a preferred choice for industries like aerospace and automotive due to its excellent processing properties. However, during the machining of Ti-6Al-4V, substantial heat is generated due to its relatively low elastic modulus and thermal conductivity. This excess heat accelerates tool wear and can lead to tool failure. In recent years, AlTiN and AlCrN hard coatings have become widely used for cutting tools. Machining difficult-to-cut materials has become a trend, and to enhance the properties of the coatings for processing these materials. Appropriate amounts of Boron (B) elements can be added to improve hardness, toughness, thermal stability, and wear resistance of the AlTiN and AlCrN coatings. The addition of Boron (B) atoms promotes the formation of a nano-composite structure, which includes AlTiBN and AlCrBN solid solutions, surrounded by an amorphous BN phase. In this study, the influence of AlTiBN/AlCrBN coatings with different multilayer architectures on the wear behavior and cutting performance of the carbide cutting tools was investigated in machining of Ti alloys. The multilayer thickness and alloy content of the deposited coating were correlated with the evaporation rate of cathode materials. Glancing angle X-ray diffraction was used to characterize the microstructure and phase identification of the films. The microstructure of the deposited coatings was characterized by using a field emission scanning electron microscope (FESEM) and a high-resolution transmission electron microscope (HRTEM) equipped with energy-dispersive X-ray spectroscopy (EDS). A Rockwell indentation tester and a scratch tester were used to evaluate the adhesion strength between the coating and the substrate. The coating hardness and the elastic modulus were measured by nanoindentation. The design of multilayered AlTiBN/AlCrBN coatings is anticipated to inhibit the grain growth, and leads to grain refinement effect, which expected to increase the mechanical properties and cutting performance of coatings.

Tribology and Mechanics of Coatings and Surfaces
Room Town & Country B - Session MC3-2-ThM

Tribology of Coatings and Surfaces for Industrial Applications II

Moderators: Nazlim Bagcivan, Schaeffler Technologies GmbH & Co. KG, Germany, **Stephan Tremmel**, University of Bayreuth, Germany, **Martin Welters**, KCS Europe GmbH, Germany

8:00am **MC3-2-ThM-1 Interactions between Coatings/Surfaces and Lubricants: How to Manage the Tribochemical Wear in ZDDP-lubricated DLC Coatings?**, ***Maria Isabel De Barros (maria-isabel.de-barros@ec-lyon.fr)***, Laboratory of Tribology and System Dynamics Ecole Centrale de Lyon, France **INVITED**

Solid and liquid lubrication play a key role in reducing energy consumption and wear behaviour of mechanical parts. Regarding the high importance of Diamond-Like-Carbon (DLC) coatings in the transportation and wind energy sectors, it is necessary to investigate their friction and wear mechanism in lubricated conditions. For these DLC films, mainly carbon and hydrogen are known to have a high chemical inertia under static conditions towards their environment. But in a lubricated sliding contact, a kind of complex "chemical reactor" is operating under severe dynamic conditions. The reason based on mainly the possibility of breaking of C-H and C-C bonds under the effect of shear, which leads to the emergence of C° free radicals or "dangling bonds" on the rubbed surface of thecoatings. A wide variety of tribochemical reactions with the lubricant molecules follow at the contact asperities, areas subjected to high pressure.

It was recently showed that very hard sp3-hybridized hydrogen-free amorphous carbon (ta-C) lubricated in the presence of the ZDDP (zinc-dialkyldithiophosphate) additive shows significant wear, associated with the absence of phosphate- type tribofilm formation. The wear is related to a preferential reactivity between the sulphur atoms released by the ZDDP and the reactive carbon atoms formed on the ta-C surface. In contrast, the softer DLCs, hydrogenated DLC and non-tetrahedral a-C, show much lower wear rates and the formation of ZDDP-derived tribofilms with higher coefficients of friction. Thus, the decomposition of ZDDP appears to be governed primarily by contact pressure at the bearing asperity scale, but the surface and subsurface sulphur transport that leads to tribofilm formation or significant wear and, results in a change in material properties at the nanoscale, depends on both stiffness and surface chemistry. In this context, the understanding of the reactivity of different DLC coatings representative of the variety of DLC found on the market and industrial applicationsin terms of H content and mechanical properties, and the interplay of mechanical and chemical contributions is of primary importance. Ultimately, itis targeting to optimize "multi-functional DLC surfaces", combining mechanical properties and surface chemistry, to better control their friction and wear performances in application.

8:40am **MC3-2-ThM-3 Coating of Plastic Parts with Tetrahedral Amorphous Carbon for Wear Protection Using Laser-Arc Technology**, *B. Gebhardt, M. Holzherr, M. Kopte, H. Pröhl, R. Seifert,* ***Marc Tobias Wenzel (Wenzel.MarcTobias@vonardenne.com)***, VON ARDENNE, Germany; *F. Kaulfuß, F. Härtwig,* Fraunhofer IWS, Germany

Laser-arc technology is a well-established method to produce hydrogen free ta-C coatings. Such coatings are known to provide outstanding wear properties and reduced friction for a variety of applications like piston rings, tappets, motorcycle chains or cutting tools.

Plastic components are ubiquitous in industry and everyday life. The use of plastic parts instead of metal components enables savings in cost, weight, and energy. Yet, coating of plastics with ta-C poses challenges due to the low hardness and limited temperature resistance of the substrates.

Injection molded plastics are shown to be suitable for ta-C coating on industrial scale using laser-arc. Reinforced and non-reinforced parts of PA12 and PEEK have been coated and investigated. The promising coating variants were transferred to a gear system. In dry operation the ta-C coating increased the lifetime by a factor of five.

In addition, we will present an up-scaled laser-arc module allowing increase of productivity and reduction of coating cost.

9:00am **MC3-2-ThM-4 Investigation of the Mechanical and Tribological Properties of TiBCN Thin Films**, ***Cennet Yıldırım (cennetyildrm@gmail.com)***, Turkish Energy, Nuclear and Mineral Research Agency – Boron Research Institute / Istanbul Technical University, Türkiye, Turkey; *Ö. Kısacık, H. Doyuran, C. Eseroğlu,* Turkish Energy, Nuclear and Mineral Research Agency – Boron Research Institute, Türkiye, Turkey; *E. Kaçar,* Hakkari University, Türkiye, Turkey

Recently, coatings with a nanocomposite structure have become increasingly significant compared to monolithic coatings, owing to their advantageous properties such as high hardness and wear resistance. Particularly, the impact of boron and carbon on coating hardness has led to a growing application of boronrich and carbide-rich nanocomposite coatings. In the scope of this study, nanocomposite films with a TiBCN structure were produced on M2 HSS using titanium and B_4C magnetron targets. To enhance adhesion on steel surfaces, Cr-CrN bond layers were deposited using the cathodic arc physical vapor deposition (CaPVD) technique, and films with different compositions were subsequently produced by varying the power of titanium and B_4C targets. Cross-sections of the produced films were examined using electron microscopy, and surface morphologies, as well as film thicknesses, were determined. Phases formed were analyzed by XRD, Raman, and FTIR, and changes in bond structures and depth profiles were identified using XPS. The hardness of the produced films was measured using the nanoindentation method, and adhesion was examined through scratch tests. Wear behaviors against alumina balls were investigated at different temperatures ranging from room temperature to 600 °C. Wear volumes were determined using optical profilometry, and wear rates and friction coefficients were calculated. The formed tribo-films were characterized using XPS analysis. It was observed that the amount of boron and carbon incorporated into the films had an influence on hardness and wear behaviors.

9:20am **MC3-2-ThM-5 Investigating the Influence of B, C, and N on the Tribo-mechanical Properties of the Chemically Complex TiSiBCN Thin Film using Design of Experiments**, *W. Tillmann,* ***Julia Urbanczyk (julia.urbanczyk@tu-dortmund.de)***, *A. Ebady, A. Thewes, G. Bräuer, N. Lopes Dias,* TU Dortmund University and TU Braunschweig University, Germany

TiSiBCN thin films show promising properties like high hardness and improved tribological behavior. Adjusting the chemical composition can tailor the properties of these thin films. To investigate this influence, usually one element is varied. However, the interplay and influence of especially the light elements B, C and N on the tribo-mechanical properties of chemical complex TiSiBCN thin films remain unclear. Therefore, a Design of Experiment using a Central Composite Design (CCD) was employed to investigate the influences of these light elements on the tribo-mechanical properties of TiSiBCN thin films. TiSiBCN with varying chemical compositions were grown in a magnetron sputtering process by adjusting the cathode power of $TiB_2/TiSi_2$ composite targets and the gas flow rates of C_2H_2 and N_2.

X-ray diffraction (XRD) analysis revealed crystalline phases based on Ti, TiN, TiC, and TiB_2, with varying degrees of crystallinity dependent on the chemical composition. Depending on the chemical composition, the TiSiBCN thin films demonstrate a broad spectrum of mechanical properties, with hardness and elastic modulus ranging from 20.2 to 39.7 GPa and from 222.3 to 405.0 GPa, respectively. Notably, the B content significantly affects the mechanical properties, with the highest hardness and elastic modulus observed at 46.0 at.–% B. In tribometer tests against Al_2O_3 under dry friction at room temperature, the TiSiBCN thin films also exhibit a broad spectrum of tribological properties, with the coefficients of friction (CoF) between 0.62 and 0.89 and wear rates between 6.4×10^{-5} and 12.2×10^{-5} mm^3/Nm. The lowest CoF of 0.62 with a wear rate of 7.7×10^{-5} mm^3/Nm is obtained for TiSiBCN with high amounts of 31.1 at.-% C and 33.5 at.–% N, while high 31.7 at.–% C and low 11.2 at.–% N contents favor the lowest wear rate of 6.4×10^{-5} mm^3/Nm with a CoF of 0.74. The tribological results reveal the significant influence of C and N on friction and wear, with TiSiBCN displaying reduced friction and wear tending to have lower hardness. Consequently, TiSiBCN thin films with either high hardness or enhanced friction and wear performance are attainable by adjusting the chemical composition.

Depending on the application requirements, the content of the light elements is decisive for the properties of TiSiCN thin films. The CCD provides insights into the intricate interplay between the chemical composition and tribo-mechanical performance of TiSiBCN. Adjusting the concentrations of B, C, and N within TiSiBCN is crucial for tailoring the tribo-mechanical behavior to meet the specific requirements of applications.

9:40am **MC3-2-ThM-6 Effect of Alloy Modification on the Wear Protection Coatings Made of Ni- and Co-Based Materials and Surface Machinability via Ultrasonic Milling Process***, Maraike Gräbner (maraike.graebner@tu-clausthal.de),* Clausthal University of Technology, Institute of Welding and Machining, Germany*; M. Giese,* Federal Institute for Materials Research and Testing, Germany*; K. Treutler,* Clausthal University of Technology, Intitute of Welding and Machining, Germany*; S. Lorenz, V. Wesling,* Clausthal University of Technology, Institute of Welding and Machining, Germany*; D. Schröpfer, T. Kannengießer,* Federal Institute for Materials Research and Testing, Germany

The development of technologies for climate-neutral energy generation is an important contribution to the reduction of greenhouse gases, whereby the efficient use of material systems is a key factor. Wear-resistant coatings are required for highly efficient and economical steel components in plant, process and power plant engineering to withstand the high corrosive, tribological, thermal and mechanical stresses. Co alloys are utilized as wear protection coatings for steel components that are customized to the specific application. The research area of interest is the substitutability of Co alloys with Ni-based wear protection systems. This research endeavour considers the price and supply uncertainties as well as the escalating demands on corrosive load-bearing capacity at elevated temperatures.

The wear-resistant alloys NiMoCrSi (Colmonoy C56) and CoMnCrSi (Tribaloy T400) have been modified through various alloying additions and subsequently applied to a carbon-manganese steel S355 using the Plasma Arc Transferred Arc (PTA) welding process. The influence of the alloying additions on the microstructure as well as on the formation of the hard phases of the build-up welds is compared. The inclusion of the alloying element Nb, for instance, results in the formation of a more refined hard phase and reduces the machining force required for the C56 and T400. The incorporation of Al results in an enhancement of the cutting forces for the C56, as the hard phases exhibit more needle-like structures. Al reduces the cutting forces of the T400. The wear potential of the modified build-up welds of the C56 and T400 is also being examined In the industrial sector, there is a growing demand for functional surfaces of superior quality. Therefore, it is imperative to ensure the machinability of the wear protection layers to achieve clearly defined contours. The machinability of the build-up welds is investigated using ultrasonic milling. The optimization of the demanding machining conditions through alloy modifications of the Co- and Ni-based alloys without impairing the wear protection potential and using the ultrasonic-assisted milling process is a joint project of BAM and ISAF at Clausthal University of Technology (Fosta P1550/IGF 21959 N).

10:20am **MC3-2-ThM-8 An Alternative Thermal Route to Improve an Aluminum Alloy Mechanical and Tribological Properties through Deposition of NiP Coating***, R. Davies,* Pontificia Universidade Católica do Parana, Brazil*; M. Soares,* Universidade Tecnologica Federal do Parana, Brazil*; F. Amorim, P. Soares, C. Neitzke,* ***Ricardo Torres (ricardo.torres@pucpr.br)****,* Pontificia Universidade Católica do Parana, Brazil

This research aims to improve the main limitations of aluminum alloys: mechanical and wear resistance through the deposition of the nickel-phosphorus (NiP) coating. Due to the natural formation of a dense oxide layer on the aluminum surface, the NiP deposition process often becomes more costly. Furthermore, it is common for NiP coatings to undergo a post-heat treatment to increase hardness due to crystallization and Ni3P precipitation and further increase adhesion through the interdiffusion layer with the substrate. Finding an adequate interdiffusion temperature is challenging, as aluminum significantly decreases its mechanical properties. It would probably soften or even melt if subjected to an interdiffusion temperature of around 400 ºC. This work aimed to find a suitable process for depositing an autocatalytic nickel-phosphorus coating on AlCu4Ti aluminum alloy in an alternative thermal route using the aluminum typical aging temperature and time treatment to create an interdiffusion layer between NiP and aluminum substrate in a single step. The typical aging temperature of the alloy was investigated, i.e., 200 ºC, as well as the minimum temperature for the beginning of Ni3P precipitation, i.e., 250 ºC. The SEM and EDS analyses showed a NiP layer of about 40 µm, well adhered, homogeneous, without substrate exposure, and a high phosphorus content (≈ 10%) formed in the aluminum alloy surface. The interdiffusion and aging treatment condition at 250ºC/16h resulted in the highest hardness of both aluminum and NiP coating.

10:40am **MC3-2-ThM-9 High-Temperature Tribology of Cathodic Arc Deposited Altin Protective Coating***,* ***Aljaž Drnovšek (aljaz.drnovsek@ijs.si)****, P. Šumandl,* Jožef Stefan Institute, Slovenia*; Ž. Gostenčnik,* jožef Stefan Institute, Slovenia*; J. Kovač, M. Čekada,* Jožef Stefan Institute, Slovenia

The AlTiN coating is a popular hard coating for high-temperature applications. However, the most commonly used method for depositing this coating on cutting tools, cathodic arc evaporation, can result in a relatively rough surface due to micro-droplet emission. This roughness and embedded droplets in the coating matrix can significantly affect the coating's wear and friction properties.

Our objective was to assess the wear and friction properties of the AlTilN coating during both the running-in and steady-state periods under varying temperature conditions. To evaluate the performance of the AlTiN hard coating, we conducted tribological tests using a high-temperature ball-on-disc tribometer. The tests were conducted using an Al_2O_3 ball as a counter body at different temperatures. We varied the test duration at specific temperatures, ranging from 50 up to 140,000 cycles, to examine the effect of test length on the coating's wear and friction properties.

The results indicated that the coating experienced the highest wear during the room temperature test. Conversely, the wear during the running-in phase and steady-state friction were the lowest at 250°C. As the temperature increased, the wear rate rose, which we attributed to increased tribo-oxidation and fatigue caused by the high test lengths. Ultimately, the coating delaminated from the WC-Co substrate at the highest temperature. The asperities on the coating surface due to micro-droplets played a significant role in friction and wear behaviour, as they were a primary source of wear particles and the first spots of oxidation on the coating. We show that the running-in phase depends mainly on the surface condition (asperities density) at room temperature tests. In contrast, at high temperatures, the formation of a stable tribo-oxide layer in the wear track elongates this period.

We conducted detailed 3D profilometry, SEM and FIB analyses on numerous samples to determine the wear mechanisms at different stages of high-temperature wear. In addition, we conducted secondary-ion mass spectrometry (SIMS) and X-ray photoelectron spectroscopy studies to evaluate the extent of oxidation and identify different species present in the oxide layer.

The combination of these analyses allowed us to gain a comprehensive understanding of the wear mechanisms and behaviour of the AlTiN coating at high temperatures. We could identify the dominant wear mechanisms and how they evolved over test length by analysing the samples at different wear stages.

11:00am **MC3-2-ThM-10 Nanomechanical and Tribological Properties of Conversion Coatings for Railway Rolling Bearing Applications***,* ***Esteban Broitman (esteban.daniel.broitman@skf.com)****, A. Ruellan,* SKF - Research and Technology Development, Netherlands*; R. Meeuwenoord,* SKF Research and Technology Development, Netherlands*; D. Nijboer, V. Brizmer,* SKF - Research and Technology Development, Netherlands

In this study, different conversion coatings have been compared in terms of friction performance based on a single-contact oil-lubricated tribometer and on a grease-lubricated double row bearing friction test rig ran under relevant operating conditions for a railway application. Conversion layers like zinc-calcium-phosphate, manganese-phosphate and tribological black-oxide deposited onto AISI 52100 bearing steel have been compared to uncoated steel in terms of nanomechanical and tribological properties.

Our results demonstrate that the optimum tribological black-oxide conversion layer can reduce friction by more than 25% on rolling/sliding raceway contacts (ball-on-disk) and up to 80% on the sliding flange contacts (roller-on-disk), which share a significant portion of power losses in roller bearing units. Results at the bearing level demonstrate that the same optimum conversion layer can reduce the running torque by approximately 30% compared to the current products both at low and intermediate speeds relevant to intercity trains.

Reference

Broitman, E.; Ruellan, A.; Meeuwenoord, R.; Nijboer, D.; Brizmer, V. *Comparison of Various Conversion Layers for Improved Friction Performance of Railway Wheel-End Bearings*. Coatings **13** (2023) 1980.

11:20am **MC3-2-ThM-11 Impact of Fiber Orientation and Oxidation on Wear Performance of Carbon-Carbon Composites***, Hamid Mohseni (Hamid.Mohseni@prattwhitney.com), X. Fang, L. Dawag, C. Winder,* Pratt & Whitney, USA

Carbonaceous matrix reinforced with continuous or discontinuous carbon-based fibers comprises the building block of carbon-carbon (C/C) composites, one of the prominent and versatile aerospace materials. The range of constituents in C/C composites, such as carbonaceous matrix, oxidation resistant impregnation, fiber types, size, volume, orientation, weaving pattern, lay-ups, and densification method, potentially allows to achieve a unique combination of properties. This design flexibility presents numerous opportunities to achieve application-specific and novel wear resistant composites. In this investigation, two class of C/C composites each with three different fiber orientations in non-oxidized and pre-oxidized (to 5 and 10 % weight loss) condition were subjected to a reciprocating sliding wear test against Inconel 718 plates at 375°C. Whereas the average wear depth was in the 10^{-2} mm range for all the samples regardless of non-oxidized or oxidized conditions, the samples with fiber orientation perpendicular to the reciprocal sliding exhibited almost 75% higher wear. Microstructural investigation of worn and unworn samples using SEM/EDS revealed severe cracking at the interface of fibers and carbonaceous matrix that resulted in fiber pull-out and evolution of wear debris. Raman spectroscopy revealed higher D/G band intensity for the worn area that indicated higher concentration of defects induced by reciprocating sliding at the interface of perpendicular fibers and carbonaceous matrix. Presence of pre-existing porosities was found to be the precursor for wear-induced cracking and formation of wear debris. Furthermore, carbonaceous solid-lubricant transfer film was discovered on the Inconel 718 plate that justified the significantly lower average wear depth (10^{-3} - 10^{-4} mm) compared to the corresponding C/C composite samples.

12:00pm **MC3-2-ThM-13 Cr Doping Modification for Tribological Behavior of Cr/a-C Multilayer Coatings Against PEEK Under Diverse Operational Conditions***, Xiaohui Zhou (zhouxiaohui@nimte.ac.cn),* Key Laboratory of Marine Materials and Related Technologies, Zhejiang Key Laboratory of Marine Materials and Protective Technologies, Ningbo Institute of Materials Technology and Engineering, Chinese Academy of Sciences, China

Considering the increasing demands for wear-resistant materials used for various frictions with dynamic sealing parts, we employed hybrid magnetron sputtering technology to fabricate Cr/a-C multilayered coatings with and without Cr-doping modification for comparison. The tribological behaviors of coatings when paired with Polyether ether ketone (PEEK) balls was focused under different friction environments evolving atmosphere, NaCl solution, olyalphaolefin (PAO) oil, and water-in-oil (W/O). The results demonstrated that the tribological properties of all friction pairs was strongly influenced by the surrounding environment. In the atmosphere and NaCl solution, the addition of Cr promoted the formation of a-C transfer film, thereby yielding the stable and low friction characteristics. However, the dominant factor contributing to the tribological performance shifted from the coatings themselves to the PAO oil film with PAO medium. In the case of W/O solution, both the facile reactivity of Cr and the intrinsic instability of W/O mixture accelerated the existence of Cr_2O_3, which caused the more severe wear. The current observations not only identified the tribological failure mechanism of Cr/GLC coatings with and without Cr doping modifications in conjunction with PEEK counterparts, but also addressed the importance of designing and fabrication of adaptive lubricant coatings for harsh multi-environment applications.

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

Tribology and Mechanical Behavior of Coatings and Engineered Surfaces (Symposium MC) Poster Session

MC-ThP-1 Influence of Cobalt Content on the Adhesion of TiAlN and AlTiN/TiSiN Coatings on WC-Co Substrates*, **Bruna Michelle de Freitas (bruna.michelledefreitas@gmail.com)**, R. Diego Torres, D. Stolle da Luz Weiss, P. Cesar Soares Junior, C. Augusto Henning Laurindo,* Pontificia Universidade Católica do Paraná, Brazil*; F. Lacerda Amorim,* Pontificia Universidade Catolica do Paraná, Brazil

Coatings based on Ti-Al-N-Si, deposited by the physical vapor deposition (PVD) process, aim to increase the working life of machining tools. In this way, the mechanical properties of the substrate interfere with the adhesion of these coatings, reflecting on their performance. Cemented carbide, one of the most used materials for cutting tool manufacturing, is a composite consisting of tungsten carbide (WC) plus a binder phase. The binder content, typically cobalt (Co), defines its main characteristics: hardness, elastic modulus and determining its application [1]. However, the effect of cobalt composition in the cemented carbide substrate and how its variation affects the mechanical properties and adhesion of PVD coatings is not extensively investigated [2]. Therefore, the objective of this study is to evaluate the adhesion and mechanical properties (hardness (H), elastic modulus (E), and the H/E ratio) of Ti-Al-N-Si-based coatings (TiAlN and AlTiN/TiSiN) deposited by the PVD process on cemented carbide substrates with different Co concentrations (6, 8, and 10%). The surface properties of the substrates and coatings were assessed using scanning electron microscopy (SEM) with an attached energy-dispersive X-ray spectroscopy (EDS) system, X-ray diffraction (XRD), and roughness measurements. For the evaluation of mechanical properties, nanoindentation tests were performed, and adhesion was evaluated through indentation testing and scratch testing. The results show that the hardness and elastic modulus of the substrates are affected by the Co content, and the AlTiN/TiSiN coating has the highest hardness due to the presence of Si in its composition, along with higher roughness from the deposition process. In general, higher Co content in the substrate negatively affects adhesion. Through the scratch test, it was observed that the TiAlN coating has better adhesion to the substrate. Additionally, for a higher H/E ratio, there is greater adhesion for both coatings (TiAlN and AlTiN/TiSiN), and this adhesion is higher in cemented carbide substrates with low Co content.

[1] CHEN C. et al., Additive manufacturing of WC-Co cemented carbides: process, microstructure, and mechanical properties, **Additive Manufacturing**, **2**023.

[2] CHEN Y. et al., Cohesive failure and film adhesion of PVD coating: Cemented carbide substrate phase effect and its micro-mechanism, **International Journal of Refractory Metals and Hard Materials**, 2023.

MC-ThP-3 Application of *In Situ* Hydrogen Charging During Micromechanical Testing of Thin Films*, **Szilvia Kalacska (szilvia.kalacska@cnrs.fr)**,* CNRS LGF, Mines St. Etienne, France

Understanding mechanisms of deformation in thin films at the sub-micron scale is the key for designing new compositions for industrial applications. It requires the determination of strains/stresses [1], dislocation distribution [2] and the overall microstructure evolution, which is often extremely challenging. Microstructural processes during external mechanical loading are hard to observe due to the complex multiscale nature of the phenomenon. If hydrogen is present in the solid, it can cause embrittlement or enhanced cracking, when the material is subjected to stress. This would eventually lead to the reduced lifetime or critical failure of the component. Although it is known for a long time that hydrogen causes degradation of mechanical performance in metals, the microscale mechanisms remain a subject of debate. Direct H-detection within the lattice is an extremely challenging task, (continuous diffusion and outgassing issues). Microstructure observations are still mostly performed post mortem on bulk samples.

In situ H-charging is therefore essential for thin film experiments. Samples can be loaded electrochemically through the back surface [3], using a cell compatible with high-vacuum (HV) scanning electron microscopes (SEM). This way, H diffuses into the lattice from the back, avoiding contamination to the surface of interest. The developed system will be presented, focusing on the coupling of the cell with the nanodeformation stage by performing nanoindentation experiments on H-charged thin films.

References:

[1] S. Wang, S. Kalácska, X. Maeder, J. Michler, F. Giuliani, T. B. Britton, The effect of δ-hydride on the micromechanical deformation of a Zr alloy studied by in situ high angular resolution electron backscatter diffraction, *Scripta Materialia* 173 (2019) 101-105. doi: 10.1016/j.scriptamat.2019.08.006

[2] S. Kalácska, *et al.*, Investigation of geometrically necessary dislocation structures in compressed Cu micropillars by 3-dimensional HR-EBSD, *Materials Science and Engineering A* 770 (2020) 138499. doi: 10.1016/j.msea.2019.138499

[3] J. Kim, C. C. Tasan, Microstructural and micro-mechanical characterization during hydrogen charging: An in situ scanning electron microscopy study*, International Journal of Hydrogen Energy* 44 (12) (2019) 6333-6343. doi: 10.1016/j.ijhydene.2018.10.128

MC-ThP-4 Shrouding Gas Plasma Deposition Technique for Generating Wear Resistant ZnO/WS_2 Composite Films on PEEK*, **Dietmar Kopp (dietmar.kopp@joanneum.at)**,* Leobner Straße 94a, Austria

In this study, zinc oxide/tungsten disulfide (ZnO/WS_2) composite films were generated by an atmospheric pressure plasma jet (APPJ) equipped with a shrouding gas attachment on polyether ether ketone (PEEK) discs. The friction and wear properties of the ZnO/WS_2 composites sliding against 100Cr6 counterpart balls were intensively investigated by using a rotational ball-on-disk setup under dry sliding conditions at ambient room conditions. The deposited and worn coating areas were observed with a scanning electron microscope (SEM). The results indicated that low friction ZnO/WS_2 composite films have the potential to protect PEEK against mechanical motion. However, the tribological performance of the coatings are strongly dependent on the plasma-process settings (i.e. plasma current, dwell time of the powder particles in the plasma jet). In fact, there is a significant tribological improvement of the composite films in contrast to the uncoated PEEK by a factor of three.

MC-ThP-5 Wear and Corrosion Characterization and Parametric Optimization of Nb-doped Hydrogenated Diamond-like Carbon (a-C:H) Coatings*, **Ihsan Efeoglu (iefeoglu@atauni.edu.tr)**, Y. Totik, G. Gulten, B. Yaylali, M. Yesilyurt,* Atatürk University, Turkey*; R. Gunay, G. Kara, B. Altintas,* TUSAS ENGINE INDUSTRIES (TEI), Turkey

This study focuses on enhancing the wear and corrosion resistance of AISI 4130, a chromium-molybdenum alloy steel, through the application of a functional coating. Targeting various industrial uses, notably in the aerospace and automotive industries, the research aims to improve the durability and performance of AISI 4130. As the functional coating, niobium-doped hydrogenated diamond-like carbon (a-C:H:Nb) coatings were deposited using a closed-field unbalanced magnetron sputtering technique under various parameters, which were systematically optimized following the Taguchi L_9 orthogonal array method. The microstructural properties of the coatings were analyzed using a scanning electron microscope, and their crystallographic characteristics were determined using X-ray diffraction, providing a comprehensive understanding of the coating structure. To evaluate the mechanical properties, nanoindentation tests were employed, offering precise measurements of hardness and elasticity. The tribological characteristics of the DLC films were assessed using a pin-on-disc tribometer, examining their wear resistance and frictional behavior under ambient air. These comprehensive analyses reveal the a-C:H:Nb coating potential for applications requiring enhanced surface properties, combining enhanced superior tribological and corrosion performance.

MC-ThP-6 Improving Tribological Properties of Al 7075 Alloy by Two-Step Soft Plasma Electrolytic Oxidation*, **Thiago de Lima Gontarski (thiago.gontarski@pucpr.edu.br)**, G. Caetano, J. dos Santos Junior, B. Leandro Pereira, R. Diego Torres, P. Soares,* Pontifical Catholic University of Paraná, Brazil

The trend of using aluminum (Al) alloys in various industrial sectors, including naval, automotive, and aerospace, can be attributed mainly to their high specific strength. However, their relatively lower resistance to wear and corrosion could limit their applications. To address this, Plasma Electrolytic Oxidation (PEO) has emerged as an effective method to enhance the mechanical, chemical, and thermal properties of Al alloys. Hence, this study aims to evaluate the impact of sample exposure time during the PEO process on the tribological properties of the Al7075.

Specimens of Al7075 were abraded with silicon carbide sandpapers of #220 grit. Subsequently, all samples were cleansed with acetone and air-dried at ambient temperature. The PEO procedure was carried out in two stages. It employed a unipolar power source, a stainless steel counter electrode, and a silicate-based electrolyte. The first stage of PEO was the same for all samples, and it involved applying a voltage of 300V and a current of 0.5 A for one minute. The second stage was carried out with a voltage of 350V, and a current of 0.3 A, with varied exposure durations for each sample: 3, 5, 10, and 20 min. The morphology, chemical composition, and crystalline phases were characterized using Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), and X-ray Diffraction. The friction and wear properties of the samples were determined by dry linear reciprocating sliding tests in a ball-on-plate setup, using an Anton-Paar universal tribometer. An applied load of 5 N and a sliding speed of 2.5 cm/s were maintained, with a reciprocating stroke of 6 mm. The test distance was set at 40 m at 25°C and a relative humidity of 50%. SEM analyses post-PEO process revealed that surface layers of the Al substrate were characterized by numerous pores and a flattened topography. The smoothest and thickest layer was achieved with the 20-minute PEO treatment. EDS results indicated that Al and O elements were predominantly present in all coatings after various exposure times. The coefficients of friction recorded were 0.557 for the substrate, and 0.501, 0.540, 0.442, and 0.427 for the PEO treatments of 3, 5, 10, and 20 min, respectively. Concurrently, wear rates were measured at 2.84, 1.79, 2.06, 1.97, and $1.34x10^{-3}mm^3/Nm$ for the same conditions. The oxide layer with the most advantageous tribological performance was that which formed over 20 min; it withstood rupture for in excess of 1600 cycles, compared to the other layers, which failed between 700 to 800 cycles. In conclusion, the longest exposure time during the mild PEO treatment correlated with the most favorable tribological properties.

MC-ThP-8 Mechanical and Tribological Behavior of Nanolayered Sputtering MoN/MoWN Coatings*, W. Hsu,* ***Fan-Bean Wu (fbwu@nuu.edu.tw),*** Department of Materials Science and Engineering, National United University, Taiwan

This research investigated the microstructure, mechanical and tribological behavior of the molybdenum nitride, MoN, molybdenum tungsten nitride, MoWN, single layers and the nanolayered MoN/MoWN, films through reactive radio frequency magnetron sputtering, RFMS, technique. The nanolayered MoN/MoWN was prepared with fixed 50 nm MoWN building layers and MoN building layers with a thickness of 25 to 50 nm. These layers were alternately stacked to form a multilayer film with a total thickness of approximately 1 μm. The MoN single building layers presented a nanocrystalline structure while well crystalline feature was found for the MoWN layers. Through microstructure analysis, the nanolayered MoN/MoWN with a building bilayer of 25/50 nm/nm possessed continuous growth of MoWN columnar crystals along B1-MoN(111). On the contrary, the through-layer columnar grain was suppressed by the 50/50 nm/nm MoN/MoWN stacking. For mechanical and tribological behavior,the wear track of the M-50/50 multilayer film was shallower and narrower as compared to those of the 25/50 MoN/MoWN multilayer film. The superior wear resistance was attributable to the effective inhibition of continuous growth of columnar crystals by a thicker MoN building layer. Additionally, the 50/50 multilayer MoN/MoWN film exhibited larger compressive residual stress which was beneficial for hardness and tribological characteristics.

MC-ThP-10 Influence of Carbon and Boron Additions on the Wear Resistance of Fe_3Al Based Laser Claddings*, H. Rojacz, K. Pichelbauer, M. Varga,* AC2T Research GmbH, Austria*;* ***Paul Heinz Mayrhofer (paul.mayrhofer@tuwien.ac.at),*** TU Wien, Institute of Materials Science and Technology, Austria

Strengthened iron aluminides exhibit excellent mechanical properties up to 600°C, and are promising candidates to replace Co-, Cr- and Ni- rich coatings for high temperature wear protection. To improve their hardness, different strengthening mechanisms can be chosen accordingly. For this study, precipitation hardening with carbon and/or boron was used to strengthen Fe_3Al-based iron aluminides. Carbon and boron were alloyed in the range from 0-20 at.% as well as combined up to 10 at.% each to precipitate carbides, borides and carboborides to show the influence on microstructural evolution, hardness as well as wear resistance. A thorough material analysis of the developed laser claddings materials and the present phases was conducted using scanning electron microscopy, electron backscatter diffraction, hot hardness testing, nanoindentation as well as high temperature abrasion testing. Results show that the hardness can be significantly increased from ~260 HV10 (claddings without any strengthening of the Fe_3Al phase) to ~850 HV10 with boride precipitations (20 at.% B). Strengthening with carbon and boron leads to a hardness of ~670 HV10 due to the formation of carboborides as well as graphite islands (10 at.% B and 10at.% C). Alloying with carbon causes the formation of graphite lamellae as well as perovskite-type carbides $Fe_3AlC0.6$ and lower hardness of a max. of ~350 HV10 at 20 at.% C. Wear results indicate a strong dependence on the present phases, whereas a significant reduction of the wear rates can be pointed out when strengthened; comparable to classical FeCrC-based hardfacings, but with the advantage of a significantly reduced ecological impact.

MC-ThP-11 Understanding Stress in Sputter-Deposited Ti-Zr-N Alloy Films*, E. Chason,* ***Tong Su (tong_su@brown.edu)****, Z. Rao,* School of Engineering, Brown University, USA

Understanding and controlling residual stress in sputtered metal-nitride films is important because of the impact it can have on their properties.Numerous studies have quantified the stress for different systems and processing conditions and modeling has attempted to explain the stress in terms of the underlying kinetic processes.Although ternary nitride alloys are used in many applications, there is much less understanding of stress in these systems relative to the binary alloys.In this work, we present results of stress in TiZrN, TiN and ZrN at different growth rates and pressures.Comparison of the ternary alloy with the two constituent binary alloys sheds light on how the addition of a second metal element modifies the stress. The results are interpreted in terms of mechanisms that have been proposed for explaining the stress generation in sputter-deposited films. These include tensile stress due to island coalescence and compressive stress due to insertion of excess atoms into the grain boundary and the effect of energetic particle bombardment.

MC-ThP-12 Cyclic Laser Thermal Shock Resistance and Mechanical Properties of AlCrSiN/AlTiSiN Multilayer Hard Coatings*,* ***Ming-Xun Yang (u6au6vmp711@gmail.com)****,* National Formosa University, Taiwan*; C. Chang, B. Chang, Y. Chang,* National Formosa University, Taiwan

In recent years, the quaternary coatings such as AlCrSiN and AlTiSiN were excellent potential candidates for wear resistance applications due to excellent thermally stability and oxidation resistance. However, for demanding operations such as the interrupted cutting and piercing, a typical failure mechanism of these tools is cyclic thermal fatigue. Such hard coatings may suffer cracking and spalling due to the high temperature cycling impact. A hard coating with multilayer architectures possesses excellent thermal stability and oxidation resistance which is attributed to the multielement composition and multilayer structure. In this study, the cyclic thermal fatigue shock test was developed to investigate the thermal fatigue failure mechanisms of the AlCrSiN and AlTiSiN monolayered coatings and multilayered AlCrSiN/AlTiSiN coatings. The failure mechanisms of the prepared coatings under the constant and cyclic thermal shocks were compared and analyzed. Several instruments were used to analyze the characteristics of the coatings. Emission electron probe microanalyzer (EPMA) was employed for elemental composition analyses, and grazing incidence X-ray diffraction (GIXRD) was utilized to analyze the crystal structure and phases of the coatings. Field emission scanning electron microscopy (FESEM) and field emission transmission electron microscopy (FETEM) were used to observe the cross-sectional microstructures. The coatings were also subjected to scratch tests to determine the adhesion strength of the deposited coatings. A nanoindentation tester was used to measure the hardness and elastic modulus. Ball-on-disk tests were used for the tribological analyses. In this case, ball-on-disk tests were used to evaluate the wear and abrasion resistance of the coatings. The results showed that the AlTiSiN/AlCrSiN multilayer coating exhibited improved mechanical properties and wear resistance due to the multilayer structure. SEM observations of the surface morphology of the thin films after single and 100 cycles of laser irradiation revealed that the AlTiSiN/AlCrSiN coatings showed less laser-induced thermal cracks, indicating excellent thermal fatigue performance of the multilayer coatings compared to respective monolayer coatings.

MC-ThP-13 Fracture Toughness of Borided AISI 1045 Steel with a Diffusion Annealing Process*, A. MENESES AMADOR,* Instituto Politécnico Nacional, Mexico*; A. OCAMPO RAMIREZ,* Universidad Veracruzana, Mexico*; A. Ballesteros-Arguello, J. Ceron Guerrero,* ***FELIPE NAVA LEANA (felnaval@gmail.com)****,* Instituto Politécnico Nacional, Mexico

A numerical-experimental study of the fracture toughness of iron borides obtained by cross-sectional scratch test was carried out. The iron borides were formed on an AISI 1045 steel. The powder-pack boriding process was developed at 1000 °C and 4 h of exposure time. The diffusion annealing process was performed on the borided steel at a temperature of 1000 °C with 4.5 h of exposure using SiC powder.The scratch tests were carried out on the cross-sections of borided material using a CSM Revetest-Xpress commercial equipment with a Vickers indenter. The scratch distance was of 1.2 mm with a load range from 5, 10 and 15 N. The applied loads and damage observed at the samples surface (with half cone geometry) were used to estimate the fracture toughness of the system. The numerical model based on the finite element method of the cross-sectional scratch test was developed considering the same test conditions. The numerical results were used to establish parameters employed in the methodology of fracture toughness by cross-sectional scratch testing.

MC-ThP-14 Influence of Cu Addition on Microstructure, Mechanical and Tribological Properties of Fe/NbC Coatings Produced on Tool Steel Using Laser Surface Alloying*, **Dariusz Bartkowski (dariusz.bartkowski@put.poznan.pl)**,* Poznan University of Technology, Poland*; P. JURČI,* Slovak University of Technology in Bratislava, Slovakia*; A. BARTKOWSKA,* Poznan University of Technology, Poland*; P. GOGOLA,* Slovak University of Technology in Bratislava, Slovakia*; D. PRZESTACKI, A. PATALAS, M. ROGALEWICZ, P. POPIELARSKI, P. SIWAK,* Poznan University of Technology, Poland

The work presents the influence of manufacturing parameters on properties of Fe/NbC composite coatings metallurgically bonded with tool steel substrate during the laser surface alloying. The Fe/NbC coatings were produced in two stages. In the first stage, an pre-coat in the form of paste was applied on substrate. In the second stage, pre-coat was remelted using a 3 kW diode laser beam. Three laser beam powers: 350 W, 500 W and 650 W were used. Various variants of powder mixture to produce pre-coats were applied: 100% NbC, NbC/4%Cu, NbC/8%Cu and NbC/12%Cu. The amounts of individual components were determined by weight. The main goal of the research was to check the possibility of producing composite coatings reinforced with NbC particles. The influence of copper addition on the properties of these coatings was investigated. Microstructure, microhardness, chemical composition tests using energy-dispersive X-ray spectroscopy and phase composition tests using the X-ray diffraction method were carried out. For tribological tests the Ball-on-Flat sliding wear method was used. The Fe/NbC coatings were analyzed both for adhesion using bronze balls and for abrasion using two types of balls - steel and tungsten carbide. The pressure forces of individual balls were selected in such a way that the average contact stresses in accordance with Hertz's theory were 1 GPa. Basic mechanical properties were determined using the nanoindentation method. It was found that it is possible to produce composite coatings metallurgically bonded to steel substrate, in which the reinforcing phase is NbC, and the role of the matrix is played by iron taken from the substrate or a mixture of iron and copper introduced with the pre-coat. It was found that some of the NbC particles were melted completely and released in situ in the matrix. However, some of these particles remain in the primary form. It was found that increasing the copper content in pre-coat leads to decrease in the microhardness of the coating matrix. In the case of coatings without copper, the hardness of 800 HV was achieved, and in the case of the addition of 12% Cu, the maximum hardness was 600 HV. However, this was the hardness of matrix without taking into account the reinforcing particles in the form of primary NbC carbides. Similar relationships occurred in the case of wear resistance. In this study design of experiment methods, which made it possible to determine the significance of the impact of researched input variables of the manufacturing process on the properties of obtained coatings were used.

MC-ThP-15 Microstructure, Mechanical and Tribological Behavior of Fe/Mo2c Coatings Produced by Laser Surface Alloying on Tool Steel*, D. BARTKOWSKI, A. BARTKOWSKA,* Poznan University of Technology, Poland*; P. JURČI, M. KUSY,* Slovak University of Technology in Bratislava, Slovakia*; D. PRZESTACKI, **Michał ROGALEWICZ (michal.rogalewicz@put.poznan.pl)**, P. SIWAK, P. POPIELARSKI,* Poznan University of Technology, Poland

The work presents the influence of manufacturing parameters on the microstructure and properties of metallurgical Fe/Mo_2C composite coatings produced in the laser surface alloying process. Tool steel was used as the substrate. The coatings were produced in two stages. In the first stage, pre-coats based on Mo_2C powder were produced. Thicknesses of these pre-coats were 150 μm, 250 μm and 350 μm. In the second stage, pre-coats were remelted with steel substrate using a 3 kW diode laser beam. A constant laser beam scanning speed of 3 m/min and three laser beam powers: 500 W, 700 W and 900 Wwere used. The aim of the studies was to determine the possibility of producing coatings with a composite microstructure, where the matrix will consist of iron from the substrate and Mo_2C particles will be the reinforcing phase. Microstructure tests using a scanning electron microscope, microhardness tests, chemical composition tests using the energy-dispersive X-ray spectroscopy method and phase composition tests using the X-ray diffraction method were carried out. Basic mechanical properties were checked using nanoindentation. Wear resistance tests were also carried out. To determine friction properties Amsler type method was used. Hardened steel was used as a counter specimen. It was found that it is possible to produce composite coatings metallurgically bonded to the substrate, in which the reinforcing phase is Mo_2C and the role of the matrix is played by iron from the steel substrate. This work presents the model for producing this type of coatings. It was found that the laser beam power and the thickness of pre-coats have influence on obtained Fe/Mo_2C coatings properties. Thanks to the use of design of experiments methods, the significance of the influence of the tested production parameters on the properties of the obtained coatings was determined. It was found that increasing the laser beam power leads to a decrease in the hardness of Fe/ Mo_2C coatings. The most favorable hardness and wear resistance are obtained for coatings produced using pre-coat thickness of 150 μm and 250 μm. Further increasing the thickness of the pre-coat results in deterioration of mechanical and operational properties. This is related to the reduced amount of matrix material in the coating and thus the lack of a matrix binding the Mo_2C particles.

MC-ThP-16 Mechanical Properties, Microstructure and Tribological Behavior of TaC Coatings Produced Using Laser Surface Alloying on Monel®400 Alloy*, A. BARTKOWSKA, D. BARTKOWSKI,* Poznan University of Technology, Poland*; P. JURČI,* Slovak University of Technology in Bratislava, Slovakia*; D. PRZESTACKI, **Paweł POPIELARSKI (pawel.popielarski@put.poznan.pl)**, P. SIWAK, A. MIKLASZEWSKI, M. ROGALEWICZ,* Poznan University of Technology, Poland

The work presents the characteristics of metallurgical TaC coatings produced on the single-phase Monel®400 alloy using the laser surface alloying method. The coatings were produced in two stages. In the first stage, pre-coats based on TaC powder were produced. They had a thickness of 260 μm +/- 10 μm. In the second stage, pre-coats were remelted with a Monel®400 alloy substrate using a 3 kW diode laser beam. A constant scanning speed of laser beam: 3 m/min and three laser beam powers: 350 W, 450 W and 550 W were used. Microstructure tests using a scanning electron microscope and microhardness tests were carried out. Additionally chemical composition tests using the Energy-dispersive X-ray spectroscopy method and phase composition study X-ray diffraction were investigated. The basic mechanical properties of the produced coatings were determined using nanoindentation. Tribological tests were also carried out. Test method for linearly reciprocating Ball-on-Flat sliding wear was used. The produced coatings had a composite microstructure with a clearly separated TaC reinforcing phase from the nickel-copper based alloy matrix. It was found that it is possible to create composite coatings on the Monel®400 alloy while simultaneously implementing this alloy from the substrate to the coating as a matrix. Based on the conducted studies, it was found that the increase in the laser beam power has influence on decrease of content of the reinforcing phase. The microstructure influences both microhardness results and wear resistance. In the matrix area microhardness ranging from 400 HV to 700 HV were obtained, but much higher values (even over 1600 HV) were observed in the areas significantly changed by the melted TaC carbide. The significance of influence of laser beam power on individual mechanical properties was determined. It was taken into account that increasing the power of the laser beam contributed to a change in the microstructure, including the complete melting of some primary TaC particles and their separation as secondary carbides.

MC-ThP-17 Influence of Differently Manufactured TiAl Targets on the Structural and Tribo-Mechanical Properties of Arc-Evaporated TiAlN Thin Films*, **Finn Ontrup (finn.ontrup@tu-dortmund.de)**, N. Lopes Dias,* TU Dortmund University, Germany*; D. Stangier,* Oerlikon Balzers Coating Germany GmbH, Germany*; N. Denkmann, A. Meijer, S. Jaquet, J. Debus, D. Biermann, W. Tillmann,* TU Dortmund University, Germany

The cutting performance and service life of WC-Co milling tools can be improved through the application of TiAlN thin films. Among the different Physical Vapor Deposition (PVD) methods, arc evaporation is widely employed to coat cutting tools due to its high deposition rate and excellent adhesion of the thin films. The TiAl targets are typically produced either by smelting or powder metallurgical methods, depending on the Al/Ti ratio. Smelting is commonly employed for Al/Ti ratios up to 1, while powder metallurgy becomes necessary for Al/Ti ratios exceeding 1. The impact of different deposition parameters on the arc evaporation process is well studied, while little is known about the influence of the target manufacturing route on the resulting tribo-mechanical properties.

Differences in the manufacturing route manifest themselves in the microstructure of the targets, which in turn results in different arc conditions at the target surface during deposition and may also cause a droplet formation. Therefore, TiAlN thin films are deposited by advanced plasma assisted (APA) arc sources, using smelting and powder metallurgical TiAl targets at three distinct working pressures (8500, 5000 and 3000 mPa).

The correlation between the target and the deposition conditions is analyzed with respect to quantity and type of droplets within the thin film. These measurements are supplemented by 3D optical roughness measurements of the thin film surface. Scanning electron microscopy SEM is used to analyze the morphology and topology, while the chemical composition is determined by microprobe analysis as well as tip-enhanced Raman scattering. Additionally, the influence of the different target types on their phase composition is evaluated employing X-ray diffraction. The hardness and the coefficient of friction are determined to examine the tribo-mechanical behavior of the thin films on cemented carbide substrates. These comprehensive analyses provide insights into the relationship between the target manufacturing route and the resulting structural, physico-chemical and tribo-mechanical properties of TiAlN thin films. The results will enhance the fundamental understanding of the interaction between target type and thin film properties of arc-evaporated TiAlN.

MC-ThP-18 Formation of TiB_2/TiB Layers on Ti_6Al_4V Alloy: Adhesion and Wear Resistance*, J. Escobar-Hernández, G. Rodríguez-Castro, J. López-Rodríguez, A. Meneses-Amador, A. Cruz-Ramírez,* ***T. N. Cabrera-Yacuta (tcabreray1800@alumno.ipn.mx)****,* Instituto Politécnico Nacional, Mexico

Adhesion and wear of Titanium borides (TiB_2 and TiB) formed on Ti_6Al_4V alloy wereevaluated by scratch tests. The powder-pack boriding at 1100 °C during 10, 15 and 20 h under inert argon atmosphere was applied to Ti6Al4V alloy. TiB2 and TiB phases were identified on the surface of Ti alloy with maximum thicknesses of 9.3 and 8.6 µm, respectively. By instrumented indentation, hardnesses were determined around 42 GPa for TiB2 and 24 GPa for TiB. The Rockwell C tests classify the adhesion of the systems as acceptable (HF3 type), regardless of the layer thicknesses. While in the scratch tests, the behavior of the coefficient of friction increased from 0.2 to 0.6 as the indenter penetrates and the damage mechanisms identified were hertzian cracks, chipping, and spallation. After, the multi-pass scratch test was employed to evaluate wear behavior at subcritical loads (40 and 50% of chipping critical load) applying 100 cycles. According to the results, the friction coefficient was not affected by the titanium boride thicknesses, but the wear rate reached its maximum reduction at 20 h of boriding with 3,7 x$10^{-3}$$mm^3$/N*m.

MC-ThP-19 Effect of MoS_2 Additive on Corrosion and Tribocorrosion Property of Plasma Electrolytic Oxidation Coating on Titanium*, N. Zheng,* National Taiwan University of Science and Technology, Taiwan*;* ***Chun-Wei Chang (yiwenz988@gmail.com)****,* Ming Chi University of Technology, Taiwan, Republic of China*; C. Wang,* National Taiwan University of Science and Technology, Taiwan*; C. Tseng,* Ming Chi University of Technology, Taiwan, Republic of China

Plasma electrolytic oxidation (PEO) technology as a novel and attractive surface engineering process has been widely used for preparation of functional oxide coatings on light alloys such as aluminum, magnesium, zirconium, and titanium. In this study, we fabricated the MoS_2 decorated composite oxide layers on pure titanium by using PEO treatment under pulsed DC power with unipolar mode in alkaline phosphate- and aluminate-based solutions with 0~3 g/L MoS_2 nanoparticle additions. The influence of MoS_2 nanoparticle addition on the microstructure, mechanical property, corrosion resistance and tribocorrosion behavior of PEO composite coating on pure titanium was investigated by X-ray diffraction (XRD), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), field-emission electron probe microanalysis (FE-EPMA), surface profilometry (α-step), scratch adhesion testing, pin-on-disc wear testing and potentiodynamic polarization measurement in 3.5 wt% NaCl solution. The experimental results obtained from scratch adhesion testing and potentiodynamic polarization measurements show that PEO composite coating with 2.5 g/L MoS_2 nanoparticlesaddition exhibits optimal adhesion strength and corrosion resistance. Furthermore, the results of XRD and SEM-EDS indicate that regardless of the presence or absence of MoS_2 nanoparticle additives, the PEO composite coatings on pure titanium are primarily composed of aluminum titanate (Al_2TiO_5) and rutile-phase titanium dioxide (TiO_2). The FE-EPMA data reveal that MoS_2 particles are mainly well distributed at the interface between the PEO coating and pure titanium substrate.The tribocorrosion behavior of MoS_2 nanoparticle decorated PEO composite coatings was carried out by potentiodynamic polarization measurement in 3.5 wt% NaCl solution under wear mode. As similar to static potentiodynamic polarization measurement, the PEO composite coating with 2.5 g/L MoS_2 nanoparticles addition also displays optimal tribocorrosion resistance in this study. In summary, the adhesion strength, wear resistance and corrosion/tribocorrosion resistance of Al_2TiO_5–rutile TiO_2 composite coating on pure titanium can be improved by increasing MoS_2 nanoparticles addition. The optimal concentration of MoS_2 additive is 2.5 g/L.

MC-ThP-20 An Improved Statistical Nanoindentation Methodology*,* ***Esteban Broitman (esteban.daniel.broitman@skf.com)****, Y. Kadin, P. Andric,* SKF - Research and Technology Development, Netherlands

The principle of statistical nanoindentation proposed ca. 2006 is based on performing a relatively large number (many hundreds/few thousands) of single indentation tests in a grid and analyzing the indentation elastic modulus and hardness with statistical methods. Many authors have claimed that this method can be used to study composite materials, showing that mechanical properties, number, and volume of the different composite phases could eventually be deduced and predicted by only using the nanoindentation technique.

In this presentation, we first review the previous work done in statistical nanoindentation by different researchers, highlighting the main problems that have been encountered and possible proposed solutions. In the second part, we study and report the statistical nanoindentation of three composite model samples, in the form of a soft Al2124 matrix embedded with hard SiC particles. We propose a novel heuristic wavelet technique to filter the measurement noise from the raw nanoindentation data as an attempt to obtain a more robust statistical nanoindentation methodology. Furthermore, a Finite Elements modeling will be used to analyze the response of the nanoindenter regarding the position of the hard particles. Our modeling will show many mistakes made by authors in previous publications. Finally, we will introduce results on bearing steels. Hardness histograms generated by Statistical Nanoindentation will demonstrate unique characteristics (fingerprints) for the different analyzed steels.

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MC-ThP-21 Adhesive Strength and Diffusion Model for Borided Ti6Al4V Alloy*, A. MENESES AMADOR, G. RODRIGUEZ CASTRO,* Instituto Politécnico Nacional, Mexico*;* ***DIEGO ALONSO BAUTISTA ALVAREZ (d.bautistaalvarez@gmail.com)****,* INSTITUTO POLITECNICO NACIONAL, Mexico*; I. CAMPOS SILVA,* Instituto Politécnico Nacional, Mexico

In this work, the Ti6Al4V alloy was hardened by powder-pack boriding process at temperatures from 1000 to 1100 °C for 10, 15 and 20 h. The boride layers formed on the Ti6Al4V alloy were examined by scanning electron microscopy and X-ray diffraction, while their mechanical behavior was evaluated by Berkovich nanoindentation and Hertzian contact. The growth kinetics of the boride layer formed on Ti6AL4V alloy was investigated based on the boron activation energy. The boride layer consisted of an outer TiB_2 layer and a TiB whiskers sub-layer. A diffusion model was proposed to estimate the boron activation energies in the TiB_2 and TiB layers, where values of 91.2 and 146.5 kJ mol^{-1} were obtained considering the experimental data of the thicknesses of the boride layers. Young's modulus and hardness were at the range of 350-400 GPa and 20-25 GPa for TiB and TiB_2 phases, respectively. The sample with the thinnest layer thickness showed the highest adhesive strength under Hertzian contact. Finally, finite element method was used to obtain the stress field in the layer-substrate system caused by the contact loads.

MC-ThP-22 Influence of Ti Content on the Tribological Behavior of Ti:MoS_2 Coatings Under Reciprocating Electrified Contact Conditions*,*
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The possibility of improving the durability and efficiency of heavy-loaded mechanical transmission systems by controlling external parameters is paramount in next-generation automotive powertrain and energy generation systems. Using electric current to control surface chemistry during the relative motion between two surfaces could allow electrified contacts to present reduced friction and wear, reducing energy consumption and enhancing overall system performance. The use of coatings with advanced 2D materials, such as molybdenum disulfide, may promote excellent solid lubrication under high contact stresses and pure sliding conditions. In this work, MoS_2coatings were deposited using pulsed D.C. magnetron sputtering technique with MoS_2 (purity of 99.99%) and Ti (purity of 99.9%) targets, in which changes in the power applied to the Ti target allowed the variation of the final Ti doping concentration at Ti:MoS_2 composite coatings. Reciprocating electrified tribological tests were carried out with the ball-on-plane configuration. Three electrified conditions (non-electrified, ball as cathode and ball as anode) and five Ti doping concentrations were tested. In all cases, uncoated AISI 52100 balls were pressed with a 10 N normal load against Ti:MoS_2 coated glass substrates. Tangential ball velocity was set at 3 mm/s with 4 mm stroke movement distance. Results indicated that electrification of the contact induced lower COF than the non-electrified conditions, for coatings with lower Ti concentration. Raman Spectroscopy of inner regions of the wear tracks indicated the presence of crystalline MoS_2 compared to as-deposited coatings. Coherence correlation interferometry analyses of wear tracks indicated wider tracks for the cases in which the ball was set as the cathode of the system. Also, under this condition, optical and scanning electron microscopy results showed high coating damage for all tested Ti concentrations. Results suggest that electrical potential polarity may promote selective desorption of coating ions that change surface chemistry, influencing the formation and composition of tribofilms that develop during the sliding motion and the friction and wear behavior of the tribosystem.

Author Index

Bold page numbers indicate presenter

Author Index