Tribology and Mechanical Behavior of Coatings and Engineered Surfaces

Room Royal Palm 4-6 - Session E2-1

Mechanical Properties and Adhesion

Moderators: Gerhard Dehm, Max-Planck Institut für Eisenforschung, Megan Cordill, Erich Schmid Institute of Materials Science, Ming-Tzer Lin, National Chung Hsing University, Taiwan

10:00am **E2-1-1 In-situ Mechanical Testing of Hierarchical and Gradient Nanostructures**, *J Wardini, O Donaldson,* ***Timothy Rupert,*** University of California, Irvine, USA **INVITED**

Recent innovations in materials processing have enabled the creation of nanostructured materials with unique microstructures. In this work, we focus on two promising examples: (1) nanocrystalline metals with amorphous intergranular films and (2) gradient nanograined materials, where grains size is varied near a specimen's surface. Due to the limited volumes of materials that can be made on lab scales or the geometry of typical parts, it is difficult to accurately probe the mechanical properties of these materials. In this talk, we describe the use of in-situ mechanical testing in the scanning electron microscope, with the goal of measuring important properties only from the regions of interest. We focus on properties of fundamental importance, such as yield strength, strain hardening rate, ductility, and rate sensitivity, with measurements made by microtension and microcompression of very small samples. Using these results, we revisit the design of these materials, to suggest paths for improvement in the future.

10:40am **E2-1-3 Mechanical Properties of Molybdenum Incorporated β-Ga_2O_3 Nanocrystalline Films for Extreme Environment Applications**, ***Anil Krishna Battu,*** *S Manandhar, R Chintalapalle,* University of Texas at El Paso, USA

The mechanical properties of the metal-oxide nano films are important to utilize them in extreme environmental applications. The fundamental knowledge about the mechanical behavior in relation to the microstructure and grain growth is required to predict the component life as well as performance in high temperature applications. Thin films and coatings of β-Ga_2O_3, a stable oxide of Ga, are widely used in high temperature sensors, anti-reflection coatings, and solar cells. We recently proposed and demonstrated that the life and performance of Ga coatings can be in high temperature applications by the addition of refractory metals. In this work, we performed a systematic study of the optical and mechanical properties of Mo co-doped β-Ga_2O_3 thin films. A comparative study of as-deposited and annealed samples will be presented and results will be compared to understand the effect of crystal structure, grain growth and oxidation states and how these parameters will affect the mechanical properties, such as hardness, young's modulus and durability.

11:00am **E2-1-4 Experimental Characterization and Finite Element Simulation of Damage in Thin Hard DLC Coatings**, *A Choleridis,* Ecole Nationale Supérieure des Mines de St-Etienne, France*; C Héau, M Leroy,* Institut de Recherche en Ingénierie des Surfaces, Groupe HEF, France*; S Sao-Joao, G Kermouche,* Ecole Nationale Supérieure des Mines de St-Etienne, France*; C Donnet,* Université de Lyon, Université Jean Monnet, France*;* ***Helmut Klöcker,*** Ecole Nationale Supérieure des Mines de St-Etienne, France

While DLC coated components exhibit very little wear having a reduced friction coefficient [1], under severe conditions blistering driven in service delamination can sometimes be observed.

In the present work, first perfectly adherent DLC coatings with high compressive process induced residual stresses were deposited on a M2 steel substrate and a thin under layer.

In a second step, the coated samples were tested under severe loading conditions in a novel friction test facility. The damage induced by these conditions has been characterized by post mortem SEM and FIB observations.

Finally, the novel experimental insight in coating damage allowed modeling coating delamination at a scale defined by the substrate microstructure.

Coating delamination is preceded by intense blistering. FIB cross sections on particular blisters allow analyzing blister nucleation. Blistering occurs close to the under-layer/steel interface, inside steel. After blistering, a thin layer of substrate material is attached to the under-layer. Its thickness is maximal in the center of the blister and decreases moving to its borders. EDX analyses through several zones of this film confirmed the presence of a thin iron-layer. Cracks initiate inside the M2 steel substrate, several nanometers beneath the (M2)-(under-layer) interface. The cracks then propagate towards this interface and propagation ends with interfacial failure. Carbides lead to local crack kinking.

The influence of the local microstructure (grain size, carbide distribution) on the delamination behavior has been analyzed by a mechanical (fe-based) model. This model highlights the blister interaction.

References

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Tribology and Mechanical Behavior of Coatings and Engineered Surfaces

Room Royal Palm 4-6 - Session E2-2

Mechanical Properties and Adhesion

Moderators: Gerhard Dehm, Max-Planck Institut für Eisenforschung, Megan Cordill, Erich Schmid Institute of Materials Science, Ming-Tzer Lin, National Chung Hsing University, Taiwan

1:30pm **E2-2-1 Controlling the Chemomechanical Effects in Sapphire by Ion-implantation*, Steve Bull,*** *A Yadav,* Newcastle University, UK

Modification of the chemomechanical behaviour of the surface of sapphire by ion implantation has been investigated to improve its near-surface mechanical properties (i.e. hardness). 300keV Ti^+ ions at various doses have been implanted and the concentration and damage profiles characterised using Rutherford Backscattering (RBS). At high doses ($\geq 3\times10^{16}$ Ti^+cm^{-2}), a surface amorphous layer has formed due to implantation-induced damage. Nanoindentation has been used to determine the hardness behaviour of the ion-implanted single crystal sapphire. It has been found that hardness increases at low implanted doses, which is associated with implantation-induced damage but that also chemomechanical softening of the surface is reduced due to the removal of adsorbed water layer. In-situ Raman scattering measurements demonstrate the removal of the adsorbed water at low doses, and existence of the readsorbed water at high doses. For the optimum implanted dose the water readsorption does not recur even several years after the implantation treatment. Based on this study, it is concluded that ion implantation with an appropriate ion species and dose can control the chemomechanical effect and improve the hardness of ceramics such as sapphire.

1:50pm **E2-2-2 Magnetron Sputtering of Refractory Metal Thin Films on NiTi Shape Memory Alloy Sheets*, Fabian Seifried,*** Karlsruhe Institute of Technology (KIT), Germany*; H Riedl,* Technische Universität Wien, Austria*; S Baumgaertner, H Leiste, R Schwaiger, S Ulrich, H Seifert,* Karlsruhe Institute of Technology (KIT), Germany*; P Mayrhofer,* Technische Universität Wien, Austria*; M Stüber,* Karlsruhe Institute of Technology (KIT), Germany

In this study, pseudo-elastic Ni 50.8 at.%-Ti alloy sheets of 1000 microns thickness were coated with 10 microns thick refractory metal thin films (e.g. Mo, Ta and Nb thin films), by non-reactive d.c. magnetron sputtering. These thin films were characterized with regard to their microstructure and selected mechanical properties. Microstructural characterization of the thin films included X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) analyses. Mo thin films grow in a densely packed, (110) textured b.c.c. structure with columnar grains and large crystallite size (columnar width: 280-300 nm) on the NiTi substrate. Ta and Nb thin films grow as well in a dense columnar structure; however they show X-Ray diffraction peaks of various lattice planes of the b.c.c. structures (i.e. no texture) and exhibit much smaller crystallite sizes (columnar width: 30-40 nm). Considering the specific thin film/substrate thickness ratio (1:100) of the samples, the mechanical properties of both the thin films and thin film/substrate composites were investigated on different length scales, using nano- and microindentation techniques. Nanoindentation results confirm that the values of the Young's modulus of all thin film materials are identical with the values of the related bulk metals. Microindentation revealed that the Young`s modulus of the Nb/NiTi composite is closest to that of the pure NiTi substrate. Progressive scratch tests indicate excellent adhesion of all metal thin films on NiTi. To further evaluate the elasticity of the metal films and the integrity of the composites during elastic deformation tensile tests were performed on the thin film/NiTi composites. These pre-strained samples were subsequently used for cycle fatigue testing. To evaluate the potential impact of the surface coating and the deposition process on the phase transformation behaviour of the NiTi shape memory alloy, differential scanning calorimetry (DSC) analyses were done. We will discuss in detail the correlation of microstructure and static and dynamic mechanical properties of metal coated NiTi shape memory alloys. Conclusions and recommendations will be given for a material selection of refractory thin films design as radiopaque coatings on NiTi substrates for medical applications.

2:10pm **E2-2-3 Quantitative *In Situ* SEM MEMS High Cycle Fatigue: the Critical Role of Oxygen on the Nanoscale-Void-Driven Nucleation and Propagation of Small Cracks in Ni Microbeams*, A Barrios Santos, S Gupta,*** Georgia Institute of Technology, USA*; G Castelluccio,* Cranfield University, UK*; Olivier Pierron,* Georgia Institute of Technology, USA **INVITED**

Small-scale fatigue is an active research area due to the widespread use of metallic films and micrometer-scale structures in applications such as flexible/stretchable electronics, micro and nano electromechanical systems (MEMS and NEMS), and microelectronics. This work presents an advanced small-scale, *in situ* scanning electron microscope (SEM) fatigue testing technique to characterize the fatigue behavior of electroplated Ni microbeams (with an ultrafine grained microstructure) subjected to high / very high cycle fatigue loading conditions, with and without a protective Au coating. The fatigue devices consist of MEMS microresonators that are driven at resonance inside the SEM, leading to fully-reversed loading of the microbeams at a frequency of ~8 kHz. The fatigue damage leads to a decrease of the microresonator's resonance frequency, which can be measured and used as a metric to quantify the crack growth rates. In addition, the *in situ* SEM observations allow direct evaluation of fatigue crack nucleation and propagation rates. Fatigue tests on the Ni microbeams provided direct measurement of ultraslow fatigue crack growth (average values down to 10^{-14} m/cycle) that have heretofore not been reported and highlighted strong environmental effects on fatigue lives that are three orders of magnitude longer in air than in vacuum. This ultraslow fatigue regime does not follow the well documented fatigue mechanisms that rely on large cyclic plastic zones and that are associated with larger crack growth rates (>~10^{-10} m/cycle). Instead, our study reveals fatigue nucleation and propagation mechanisms that mainly rely on room temperature vacancy condensation leading to voids, whose nucleation process is strongly affected by oxygen. The presence of a protective Au coating also increased the fatigue life in air by one order of magnitude, by delaying the crack nucleation process in the underlying Ni microbeam. The improvement in fatigue life is related to the fatigue degradation of the Au coating and its delamination from Ni once a fatigue crack reaches the interface. This work highlights significant size-dependent fatigue behaviors, whose origin will be discussed in this talk.

2:50pm **E2-2-5 Role of Microstructure on the Interface Stability of Copper Thin Films on Brittle Substrates*, Alice Lassnig,*** Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Austria*; V Terziyska,* Montanuniversität Leoben, Austria*; C Gammer,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Austria*; D Kiener, C Mitterer,* Montanuniversität Leoben, Austria*; M Cordill,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Austria

Thin ductile films on brittle substrates are widely used in different technological applications such as in microelectronics and energy sectors. Of particular interest is the stability of the interface between the two materials, since it is the weakest site determining the overall reliability of the material system. To decouple extrinsic from intrinsic size effects, a model ductile thin film on a brittle, chemically inert substrate was investigated. Therefore, 100 nm copper thin films were deposited by magnetron sputtering where the deposition process was optimized to maintain the same thin film thickness but a significant variation of the microstructure (bimodal). Further variations of the microstructures could be achieved by means of heat treatments leading to a uniform, coarse-grained microstructure. A thorough characterization of the film microstructures and interfaces was conducted by means of SEM and TEM.

To determine the adhesion, stressed overlayers were deposited, leading to the formation of spontaneous buckles with straight and telephone cord buckle morphologies. These buckles were measured by means of confocal laser scanning microscopy, allowing for the determination of the adhesion energy with the well-known Hutchinson & Suo model.

We could show that the adhesion values of the same thin film – brittle substrate system increased a factor of almost 2 with decreasing grain size but same thin film thickness.

A detailed study explaining the change of interface adhesion energy by means of a thorough characterization of the thin films will be presented, including a TEM study to reveal the delamination processes in-situ.

Abstract #3863

3:10pm **E2-2-6 Mechanical Reliability of Barrier Films for Flexible Electronics**, ***Kyungjin Kim***, *H Luo, T Zhu, S Graham, O Pierron,* Georgia Institute of Technology, USA

PECVD SiN_x thin film coatings have been developed to protect flexible electronics devices from environmental exposure. While most of the study has leaned on water vapor transport properties, the mechanical reliability during flexural deformation is another critical aspect for the coatings. Previously, we investigated the time-dependent channel crack growth behavior of silicon nitride (SiN_x) barrier films on polyethylene terephthalate (PET) substrates in humid and dry air and showed that crack growth can occur at strains that are much lower than the standard measured crack onset strains. In this work, we demonstrate the long-term time dependent fracture of PECVD SiN_x barrier films on polyethylene terephthalate (PET) and polyimide (PI) substrates by measuring crack growth rates under fixed strain as a function of time using in-situ microscopy tensile test. Our study highlights the interplay between neighboring cracks and substrate damage on the long term crack growth rates. For isolated cracks in SiN_x with PET damage, the growth rates were found to increase with time due to the loss of constraint on the SiN_{-x} channel cracks as the corresponding crack grew in the PET. For multiple cracks growing on PET, decrease in the subcritical crack growth rates was found by up to 2 orders of magnitude until reaching steady-state rates due to substrate cracking in the interacting cracks. For SiN_x on PI, crack growth rates were observed to be nearly constant due to the lack of substrate cracking in PI as compared to PET.

3:30pm **E2-2-7 Molecularly Grafted, Structurally Integrated Multifunctional Polymer Thin Films with Improved Adhesion**, *A Lassnig,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Austria*; P Smith,* Carnegie Mellon University, USA*; M Cordill,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Austria*;* ***B.Reeja Jayan****,* Carnegie Mellon University, USA

We present a novel molecular scale grafting technique using chemical vapor deposition (CVD) polymerization to enhance the adhesion of thin polymer films to various substrates. Such engineered coatings can find applications in structurally integrated batteries and sensors that can carry mechanical loads in addition to performing energy conversion functions. Conventional un-grafted polymer thin films are loosely anchored to surfaces by weak physical interactions like Van der Walls forces or physisorption, which are easily disrupted by mechanical forces. We show that grafting can deploy strong chemical bonds or chemisorption to permanently anchor the film to the substrate. Until now, direct measurements of these bonds have not been possible because the materials used are very thick (typically 10-100 µm) and are difficult to access by surface characterization methods like x-ray photoelectron spectroscopy. Also, in previous attempts these bonds were made on small (localized) scale (e.g., polymer brushes grafted on nanoparticles) and thus large spatial variations in stresses that arise under real operating conditions in a device could not be investigated.

Herein, we use a novel non-line-of-sight oxidative CVD (oCVD) polymerization technique to simultaneously graft and polymerize monomers of EDOT onto radical (reactive) sites present on a Silicon substrate, resulting in conducting poly(3,4-ethylenedioxythiophene) (PEDOT) films. This grafting improves adhesion of coatings to the substrate surface. Films without the graft spalled and delaminated in the form of spontaneous buckles, after the application of Molybdenum stressed overlayer. Similar PEDOT films with the graft only spontaneously delaminated after the same stressed overlayer was added. From the spontaneous buckling the adhesion energy of the PEDOT-Silicon interface can be evaluated using the well-known Hutchinson and Suo model. It will be demonstrated that the addition of the graft significantly increases the adhesion of the PEDOT to a Silicon substrate

3:50pm **E2-2-8 Thin-film Adhesion Characterization by Colored Picosecond Acoustics**, ***Arnaud Devos****,* IEMN UMR CNRS 8520 / MENAPiC, France*; P Emery,* MENAPiC, 41 Bd Vauban, France

Thin-film characterization is a main issue for a broad range of industrial applications related to the microelectronic industry or coatings for optics or glass industry or photovoltaics. All these technologies have in common to develop new products based on complex stacks made of various thin-film materials thinner and thinner. Thickness, elasticity, composition and adhesion at interfaces must be controlled if possible in a non-destructive way. Increased efforts are being made to develop measurement methods compatible with “real life conditions”.

This paper will present a new technique so-called the Colored Picosecond Acoustics (APiC), a unique combination of optics and acoustics, which implements a SONAR at the nanoscale using a tunable ultrafast laser. From the experimental point of view, it is a full optical setup, acoustics taking place in the sample only. Very high frequency acoustic waves are emitted and detected using ultra-short laser pulses. The acoustic waves propagate indifferently in transparent or opaque materials. These “hypersonic waves” have such a short wavelength that they suit very well the characterization of thin films, multi-layers, nanostructures and interfaces.[1-2]

In this paper, we will present some results to show useful can be the APiC technique for controlling thin-film thickness and adhesion at interface on complex samples related to various industrial objects: radio-frequency filters, thin-film solar cell, advanced mirrors, semiconductor lasers.

References:

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4:10pm **E2-2-9 Imaging Thin Film Adhesion with Picosecond Ultrasonics**, ***Allaoua Abbas****, X Tridon, J Michelon,* Neta, France

In the middle of the eighties, it has been demonstrated that femtosecond lasers are able to generate and to detect ultrasounds which frequencies can extend up to serval TeraHertz. Thanks to their very small wavelengths, these ultrasounds can perform measurement of the mechanical properties of structures with nanometric resolution. These measurements have serval application in industries which use thin films, as the photovoltaic or the electronic ones.

In this presentation, utilization of these ultra-high frequencies ultrasounds as a tool for nondestructive testing will be overlined. It will be shown how photo-generated and photo-detected ultrasounds are able to probe, without any contact, the bounding quality and the thickness of a thin metallic film deposited on a dielectric substrate. Cartographies, which illustrate the thickness inhomogeneities and the bounding quality adhesion of a 255 nm Tungsten thin film deposited on a Silicon substrate will be presented to support these points.

4:30pm **E2-2-10 Mechanical Property Evaluation of Zr-Ti- Fe Thin Film Metallic Glasses**, ***Yi-Jie Liao****,* Ming Chi University of Technology, Taiwan*; D Tseng, T Wu, M Lin,* National Chung Hsing University, Taiwan*; J Lee,* Ming Chi University of Technology, Taiwan

Recently, thin film metallic glass (TFMGs) have drawn lots of attention from academia and industries due to their unique properties and possible applications. In this study, four ternary Zr-Ti-Fe TFMGs were fabricated on Si wafer and AISI304 stainless steel disk substrates using a magnetron co-sputtering system. The power of iron target was adjusted to grow TFMGs with different Fe contents. The effects of iron content on the microstructure and mechanical properties of Zr-Ti-Fe TFMGs were discussion. The thin film metallic glass materials consisted of an amorphous structure, with an absence of any detectable peak corresponding to crystalline phases. The surface morphology of TFMGs showed a very smooth surface by the analysis of atomic force microscopy. The hardness and elastic modulus of TFMGs were analyzed by nanoindentation. Furthermore, the bulge test was carried out to determine the residual stress, elastic modulus and deformation behavior of TFMGs from the pressure–deflection curves. The influence of Fe concentration on the mechanical property and deformation behavior of ternary Zr-Ti-Fe TFMGs was discussed.

4:50pm **E2-2-11 Mechanical Properties Measurement of Submicron Ti-Ni Shape Memory Alloys Thin Films**, *T Wu,* ***Ming-Tzer Lin****,* National Chung Hsing University, Taiwan*; T Chen,* Chaoyang University of Technology, Taiwan*; T Lin,* National Chung Hsing University, Taiwan

Ti-60at%Ni thin films with thickness of 600nm were deposited on silicon nitride with and without chromium interlayer. Static and dynamic mechanical properties of these films were investigated using bulge test and observed their microstructure and crystalline phase orientation from SEM and XRD. It was found that with the adhesion of chromium interlayer the Ti-Ni films have the lowest residual stress and the highest Young’s modulus. The addition of chromium interlayer between Ti-Ni film and silicon nitrite introduces R phase transformation in room temperature. Microstructure analysis revealed that the surface trenches could be significantly reduced if the film has chromium interlayer, which indicates that chromium interlayer can minimize the stress development on Ti-Ni films during deposition. Thermal cycling stress evolution test results showed that chromium interlayer can be a buffer layer between TiNi and SiNx thermal mismatch. Moreover, the thermal cycling bulge test can be used to measure the

thermal expansion coefficient of the films. In addition, the fatigue test showed that the interlayer of Cr can enhance the fatigue strength of TiNi films.

Tribology and Mechanical Behavior of Coatings and Engineered Surfaces

Room Royal Palm 4-6 - Session E1-1

Friction, Wear, Lubrication Effects, and Modeling

Moderators: Albano Cavaleiro, University of Coimbra, Carsten Gachot, Vienna University of Technology, Nazlim Bagcivan, Schaeffler Technologies GmbH & Co. KG, Germany

8:00am **E1-1-1 Tribologically Induced Oxidation of High-purity Copper as a Function of Sliding Distance***, C Greiner, S Becker,* ***Christian Haug,*** Karlsruhe Institute of Technology (KIT), Germany

Tailoring the surface properties of a material for low friction and little wear has long been a goal of tribological research. Since the microstructure of the material under the contact strongly influences tribological performance, the ability to control this microstructure is thereby of key importance. However, there is a significant lack of

knowledge about the elementary mechanisms of microstructure evolution under tribological load.

In order to under how different elastic and plastic strains influence this microstructure evolution, both were systematically varied in high-purity copper samples. Scanning electron (SEM) and focused ion beam (FIB), electron back scatter diffraction (EBSD) and transmission electron microscopy (TEM) are applied to monitor the resulting microstructure changes.

Our results demonstrate that with increasing strain, the tribologically deformed layer grows deeper into the material. Also an influence on grain rotation and refinement mechanisms will be discussed in the talk.

These results strongly suggest that the amount of tribologically induced strain is a key elementary that needs to be addressed in future modelling of the microstructure evolution in tribological contacts.

8:20am **E1-1-2 Investigation on the Reason for Low Friction between Diamond-like Carbon Coating and Ti-6Al-4V under Fretting Conditions***,* ***Haohao Ding****, V Fridrici, P Kapsa,* Ecole centrale de Lyon, LTDS, France

Ti-6Al-4V / Ti-6Al-4V contacts submitted to fretting (for instance, in the contact between femoral stem and neck adapter in hip joints) present high adhesive wear and very high friction coefficient (around 1.0 – 1.2). Diamond-like carbon (DLC) coatings can be deposited on one of the contacting bodies to decrease friction and wear between two metallic parts in contact.

The objective of this paper is to investigate the reason for the low friction between the DLC coating and Ti-6Al-4V under fretting conditions with a cylinder-on-flat geometry contact. Flats (rough and smooth) and the cylinder (rough) are made of Ti-6Al-4V. Flats were coated with DLC coating using PACVD. The coating thickness is around 2.0 µm. The nano-hardness and the elastic modulus reach 29 ± 4.5 GPa and 236 ± 24 GPa, respectively. Tests were carried out with the normal force of 250 N and the displacement of 20 µm. The evolution of surfaces of both the DLC coating and the Ti-6Al-4V counterbody was explored.

The results indicate that the friction coefficient decreases from around 0.5 to a lower value (around 0.1) during the running-in period, corresponding approximately to the first 100 cycles. After 100 cycles, rotating the cylinder (i.e., putting in contact a new surface from the Ti-6Al-4V counterbody with the already rubbed DLC coating) leads to a new running-in period. Moving the flat (i.e. putting in contact a new surface from the DLC coating with the already rubbed Ti-6Al-4V counterbody surface) results in a continuous low friction coefficient. Those mean that the surface state of Ti-6Al-4V after running-in is the key factor to the low friction. When the DLC coating and Ti-6Al-4V are put into contact, Ti-6Al-4V is adhered to the DLC surface. When the DLC coating starts to slide, more Ti-6Al-4V is abraded and adhered to the DLC. The energy required to break the metallic bonds in Ti-6Al-4V results in the high friction. During the running-in period, the worn surface of Ti-6Al-4V is gradually covered by a tribofilm, which is mainly composed of the worn-off Ti-6Al-4V with oxidization. Carbonaceous material is also observed on the tribofilm. The tribofilm and carbonaceous material separate the Ti-6Al-4V and DLC (impeding direct contact between the 2 counterbodies) and lead to the low friction. Furthermore, the coefficient of friction is higher when the roughness of the coated flat is higher. Cracks of DLC coating are observed on rough flat, but no cracks are observed on smooth flat, because the local contact pressure between asperities from rough flat surface and rough cylinder surface is higher than that between smooth flat surface and asperity from rough cylinder surface.

8:40am **E1-1-3 Tribological and Wettability Evaluation of Magnetron Sputtered WS-C/F Coatings***,* ***Simone Pereira Rodrigues****,* University of Coimbra, Portugal*; S Carvalho,* University of Minho, Portugal*; A Cavaleiro,* University of Coimbra, Portugal

The automotive industry produces a huge amount of mechanical components daily. Namely, the ignition systems need lubrication on their assemblage step, which their excessive use is nowadays a concern because of environmental/human risks. The lubrication tools in use are often stopped for maintenance due to either deficient distribution of the lubricant or high friction phenomena. The solution for these issues is the development of functionalized surfaces addressing both low friction/longer lifetime, due to an improvement of the wear resistance, and further special water/oil wettability properties to improve the lubricant application homogeneity. Two possible surface modification approaches can be used, separately or in synergy, as solutions for those problems: (i) surface structuring by anodization processes and/or (ii) deposition of self-lubricating coatings. In this work, the latter was explored by the optimization of the deposition of self-lubricant coatings based on TMDs, alloyed with carbon and fluorine (W-S-C/F).

WS-C/F coatings were deposited by magnetron sputtering in a reactive Ar/CF_4 gas mixture, from a WS_2 target. Different F contents up to 20 at.% were achieved by varying the CF_4 flow rate. The top-view/cross-section morphologies, the chemical composition/bonding, structure and wettability of the coatings were characterized by SEM, XPS, XRD techniques and water/oil contact angle measurements, respectively. The mechanical properties such as hardness, elastic modulus were as well performed through nanoindentation procedure and the adhesion by scratch testing. The tribological performance was evaluated at room temperature (RT) and at 200 ºC at 20N load against a 100Cr6 steel ball.

The F incorporation led to higher surface hydrophilicity of the coatings, with no effect on the oil wettability, behaviour which could be related to the decrease on the surface roughness. RT tribological tests showed that averagely all tested coatings have similar friction coefficient (COF mean value=0.06), however, both pure WS_2 and the highest F-containing coatings showed a very irregular friction curve at the first 5000 running cycles. Sudden increases of COF, followed by its progressive decrease down to very low values, were observed (0.04 for pure WS_2 and 0.02 for WS-C/F coating). Tribological testing at 200 ºC (dry conditions) showed the same trend, i.e. high F-doped coating reached a COF mean value of 0.016 compared to 0.030 for WS_2 coating. This was interpreted as a beneficial effect of F on increasing the interplanar basal distance of the hexagonal WS_2 tribolayer formed in the contact, decreasing the van der Waals bonding, with the consequent COF decrease.

9:00am **E1-1-4 Tribological Properties and Oxidation Resistance of WN_x Thin Films at High Temperatures up to 500°C***,* ***Daniel Javdošňák****, J Musil, Z Soukup, R Čerstvý, S Haviar, J Houska,* University of West Bohemia, Czech Republic

The paper reports on the structure, microstructure, mechanical properties, friction coefficient µ, wear rate k and oxidation resistance of the WN_x films; here x=N/W is the stoichiometry of nitride films. The films were reactively sputtered from a W target of diameter of 100 mm on Si(100) and Steel 15330 substrates in a mixture of $Ar+N_2$ gases using an unbalanced magnetron powered by the AC pulsed power supply. The properties of sputtered WN_x films were characterized by (i) X-ray diffraction (XRD), (ii) Scanning Electron Microscope (SEM), (iii) micro–indentation testing, (iv) pin–on–disk tribometry in wide range of temperatures T from room temperature (RT) up to 500°C and (v) ellipsometry. It was found that sputtered WN_x films are polycrystalline nanocomposites composed of either a mixture of low–T α-W and high–T β-W_2N the phases at x≤0.5 or high–T β-W_2N and low–T δ-WN the phase and exhibit: (1) high values of the hardness H, effective Young's modulus E^*, elastic recovery W_e increasing with increasing x up to 34 GPa, 0.13 and 88%, respectively, (2) the friction with Al_2O_3 ball (i) increases from 0.3÷0.4 at RT to 0.8÷1.2 at 200°C and (ii) decreases to 0.5÷0.6 at 400°C and sliding distance of 1000 m, (3) the wear with Al_2O_3 ball increases from 10^{-8} mm^3/Nm at T≤200°C up to ~2.5×10^{-6} mm^3/Nm at T ranging from 200 to 400°C. The WN_x films are completely removed from the substrate at T=500°C already at sliding distances of about 350 to 600 m due to formation of the WO_x scale on the coating surface.

9:20am **E1-1-5 Correlation between Evolution of Roughness Parameters and Micropitting of Carburized Steel Surfaces under Boundary Lubrication Condition**, ***Sougata Roy***, *D White, S Sundararajan,* Iowa State University, USA

This paper investigates the correlation between the evolution of the amplitude (R_a, R_{RMS}, R_{sk} and R_{ku}) and spatial (autocorrelation length) roughness parameters and micropit initiation and propagation during rolling contact fatigue (RCF) of carburized steel samples under boundary lubrication conditions. Steel samples with three levels of retained austenite or RA (~0%, 15% and 70% as measured by micro X-ray diffraction) were prepared for the RCF study which was conducted with a rolling-sliding contact in a micropitting rig. It was observed that run-in happened within an order of 10^4 cycles for all samples and during this period, a significant decrease in R_a and R_{RMS} occurred while the correlation length increased and stabilized. The low RA samples failed due to early crack initiation and rapid crack propagation. The medium and high RA samples showed initiation and propagation of micropitting during RCF life. Micropitting initiation and propagation were captured for the mid and high RA samples using an optical profilometer which utilizes a non-contact white light interferometry technique. Micropitting trends were then correlated with different surface roughness parameters. It was observed that, if surface change due to mechanisms other than micropitting is controlled, then R_a and R_{RMS} follow the same trend with the propagation of micropitting. Skewness can be used as a parameter to predict the initiation and propagation of micropitting; significant propagation resulted in a decreasing trend of skewness (negative) and increasing trend of kurtosis. Transverse directional correlation length also was found to be in agreement with the propagation of micropitting. It was observed that the correlation length decreased as micropitting progressed. The present study shows that drivetrain industries can track correlations with surface roughness parameters to predict the long-term performance of the components under the boundary lubrication regime.

9:40am **E1-1-6 The Influence of Temperature on the Wear Mechanisms of a Cobalt-based Alloy Contact Subjected to Fretting: from an Abrasive Tribo-oxydation Process to the Glaze Layer Response**, ***Alixe Dreano***, *S Fouvry, G Guillonneau,* LTDS - Ecole Centrale de Lyon, France

Cobalt-based alloys are widely used in the aeronautical industry for their good mechanical properties and corrosion resistance at high temperature. Vibrations, or other micro-displacements, of mechanical pieces initiate fretting and then lead to wear. Wear mechanisms of cobalt-based alloys are commonly known to be dependent on temperature. Above a transition temperature TGL, wear rate decreases and becomes very low given that a compacted and oxidized layer is spontaneously created in the contact ("glaze layer"). Below TGL, wear is severe and the tribolayer is not generated. The high-temperature layer has been thoroughly studied by many authors who associated its formation to the capacity of oxidized debris to sinter and adhere to the bulk metal.

The present study was focused on the change of wear mechanisms occurring through a range of temperatures (from ambient to 600°C). A cross-cylinders cobalt-based alloy against alumina contact was subjected to gross-slip fretting. The study showed that wear, before severe-to-mild wear transition, TGL, is controlled by the continuous oxidation of the interface and is therefore strongly influenced by the operating temperature and frequency of solicitation. An analytical wear law was developed in order to describe the wear mechanism below TGL. The dependence of TGL on frequency was also outlined and associated to the capacity of debris to sinter. Finally, to completely describe the behavior of the tribocouple, the mechanical response of the glaze layer is discussed in the light of the results of in-situ compression of micro-pillars cut into the glaze layer.

10:00am **E1-1-7 Coated Surface Wear Resistance Design by Comutational Modelling**, ***Kenneth Holmberg***, *A Laukkanen, T Hakala,* VTT Technical Research Centre of Finland Ltd, Finland **INVITED**

The presentation gives an overview of Integrated Computational Materials Engineering (ICME) and the approach and techniques we have used for computational materials modelling and simulation. The focus is on meso- and microscale modelling, integrated approaches and wear related applications. The FEM modelling technique has been stretched to observe stress and fracture phenomena on microscale in thin hard diamond-like carbon (DLC) and titanium nitride (TiN) physical vapour deposited coatings and an equation was developed to show the dominating effects on potential wear failure.

A novel multiscale numerical finite element method (FEM) model was developed to integrate the layered and microstructural material features with the orientation of surface topographical features. A fractal geometry and surface voxelisation based approach were utilised to derive representative 3D topography. The simulations show the details of the main topographical orientation effects on local stresses affecting wear as they appear at a single scratch by a diamond ball and in a self-mated sliding contact between two rough surfaces. The 45º sliding direction to the grooves resulted in a mixed state of surface loading in contact during the scratch test. The complex state of stress-strain within the roughness peaks decreased the overall tensile stress state and resulted in a greater surface resistance to cracking compared to 0º and 90º directions. Model based calculations showed that the surface structure was about four times more rigid in the direction of grooving compared to the more flexible behaviour in the direction perpendicular to the grooving. This behaviour was confirmed experimentally. Modelling and simulation of a tribocontact help to understand the mechanisms that result in surface cracking, wear particle formation and wear evolution and to work out guidelines for optimal contact and surface design for best friction and wear performance tailored for specific applications. Numerical simulations can be carried out on several spatial scale levels, from nano size to macro size, by using software representing the material structure from atomic and even sub-atomic to continuum macro and component level. VTT has introduced the *VTT Propertune* approach and software to find optimal solutions to industrial material challenges worldwide (http://www.vttresearch.com/propertune).

10:40am **E1-1-9 Room and Elevated Temperature Sliding Wear Behavior and Mechanisms of a Cold Sprayed Ni-WC Composite Coating**, ***Tyler Torgerson***, *M Harris,* University of North Texas, USA*; S Alidokht,* McGill University, Canada*; T Scharf, S Aouadi,* University of North Texas, USA*; R Chromik,* McGill University, Canada*; J Zabinski,* Army Research Laboratory, USA*; A Voevodin,* University of North Texas, USA

Cold sprayed Ni-WC metal matrix composite coatings have advantageous tribological properties that have only been investigated in the literature at room temperature. This study sought to identify their elevated temperature dry sliding behavior from room temperature up to 400 °C as well as during thermal cycling with a sliding speed of 2 cm/s and a load of 2.45 N. Further characterization included the use of SEM, EDS, XRD, XPS, Raman spectroscopy, interferometry, and hardness measurements. Results indicate that an increasing temperature leads to a decrease in friction and an increase in wear. The coefficient of friction decreased from 0.41 at 23 °C to 0.32 at 400 °C, while the wear rate increased from 0.47×10^{-4} mm^3 N^{-1} m^{-1} at 23 °C to 3.67×10^{-4} mm^3 N^{-1} m^{-1} at 400 °C. This lowering of friction is attributed to the formation of a lubricious tribochemical phase in the wear track. The increase in wear is due to a combination of thermal softening and a change in the wear mechanism from adhesive to abrasive. During thermal cycling, the coating exhibited self-adaptive behavior from the high to low friction regime. The results revealed that thermal softening and tribochemical reactions that occurred at elevated temperatures slightly compromised the wear resistance while producing a lubricious tribofilm. Therefore, WC-Ni cold spray coatings are potential candidates for elevated temperature sliding wear applications.

Tribology and Mechanical Behavior of Coatings and Engineered Surfaces

Room Royal Palm 4-6 - Session E1-2

Friction, Wear, Lubrication Effects, and Modeling

Moderators: Albano Cavaleiro, University of Coimbra, Carsten Gachot, Vienna University of Technology, Nazlim Bagcivan, Schaeffler Technologies GmbH & Co. KG, Germany

2:10pm **E1-2-3 Physical Mechanisms for Nanoscale Friction of a-C:H/D Thin Films**, *F Echeverrigaray, S de Mello,* UCS, Brazil; *F Alvarez,* UNICAMP, Brazil; *A Michels,* ***Carlos Figueroa,*** UCS, Brazil

The friction forces are originated in energy dissipation events owing to the lost work of non-conservative forces. The surface structure of hydrogenated/deuterated amorphous carbon thin films in air, which plays an important role in nanoscale friction, is constituted by hydrogen and/or deuterium terminated bonds and physisorbed oxygen, nitrogen, and water molecules. In spite of friction models were well established to explain the tribological behaviour of carbon-based thin films in different atmospheres, the fundamental physical understandings of these phenomena remain open. In this work, we report the friction behaviour of a diamond spherical dome sliding on different amorphous carbon thin films containing different amounts of hydrogen and/or deuterium inspecting at the nanoscale indentation. Three different experimental setups are reported. Firstly, for samples where hydrogen was replaced by deuterium in the thin film bulk, the friction coefficient decreases with the increasing of deuterium content. Secondly, for samples where hydrogen content is increased at the surface, the friction coefficient decreases with the increasing of the ratio H/C at the surface. Thirdly, for samples where the ratio H/C was fixed, the friction coefficient increases with the increasing of the relative humidity, effect that can be tuned by an external electrical field. Finally, we discuss three different physical mechanisms describing these experimental results: dissipation effects associated with phonon coupling, van der Waals forces, and orientation of water dipoles determining the friction behaviour of a-C:H/D for the above described experimental setups.

2:30pm **E1-2-4 Relocation Profilometry of Micro-tribology Experiments of Uncoated and DLC Coated Steel**, *M Gee, J Nunn, L Crocker,* National Physical Laboratory, UK; *K Holmberg,* VTT Technical Research Centre of Finland Ltd, Finland; *L Li,* City University of Hong Kong, Hong Kong; *G Stachowiak,* Curtin University, Australia; *C Gachot,* Vienna University of Technology, Austria; ***Tony Fry,*** National Physical Laboratory, UK

Relocation profilometry was used to evaluate the damage that occurred in micro-tribology experiments on a range of DLC coated and uncoated steel samples. Three roughness conditions were tested ranging from a ground surface to a smooth surface for both the coated and uncoated materials. The effect of varying orientation of the micro-tribology experiments with the directionality of the finished surfaces. The micro-tribology experiments were carried out using single pass scratch tests using a diamond indenters. The same areas were examined with an Olympus Lext confocal microscope which gave image and height maps from each area examined. The same areas were examined before and after scratching, and the resulting pairs of height maps registered and subtracted using Image J. The true volume of damage was thereby calculated for all experiments. Friction measurements were also made. This enabled the energy dissipated in the damage formation to be calculated.

Little effect of orientation on the damage was observed. What was quite surprising was that there was also no effect of roughness on the damage that was observed.

2:50pm **E1-2-5 Microstructural Design of Self-lubricating Laser Claddings for use in High Temperature Sliding Applications**, ***Carsten Gachot,*** TU Wien, Austria; *M Rodríguez Ripoll, H Torres,* AC²T Research GmbH, Austria; *B Prakash,* Lulea University of Technology, Sweden

Nickel-based self-lubricating claddings with the addition of Ag and MoS_2 were prepared by means of laser cladding on stainless steel substrates, aiming at their implementation in metal forming applications involving demanding tribological conditions at high temperature. The novelty of our approach relies in the addition of MoS_2 with the aim achieve a uniform silver distribution within the resulting cladding by means of an encapsulation mechanism, preventing it from floating to the surface during the deposition process and being subsequently lost during surface preparation. The role of Ag and MoS_2 concentration on the encapsulation process is discussed in terms of phase composition and resulting microstructures. The tribological behaviour of the resulting laser claddings was evaluated at high temperature under unidirectional sliding. The encapsulation of Ag led to outstanding tribological properties while keeping the amount of Ag used at lower concentrations, thus increasing the economic feasibility of the claddings. The improvement in terms of both friction and wear was observed for the self-lubricating claddings compared to the reference alloy, making them good candidates for use in high temperature applications such as metal forming.

3:10pm **E1-2-6 Fretting Wear Behavior of Duplex PEO-Chameleon Coating on an Al Alloy**, ***Andrey A. Voevodin,*** University of North Texas, USA; *Y Liu,* University of Leeds, UK; *A Yerokhin,* University of Manchester, UK; *A Korenyi-Both,* Tribologix, Inc., USA; *M Lin,* University of Manchester, UK; *J Zabinski,* Army Research Laboratory, USA; *A Matthews,* University of Manchester, UK; *T Liskiewicz,* University of Leeds, UK

Plasma electrolytic oxidation (PEO) is an attractive technology for improving wear resistance and environmental protection of aluminum alloys. PEO results in the hard alumina based ceramic coatings of up to 100-150 micrometer thickness which are well adhered to the surface with morphology graded from a dense region near the coating-substrate interface to a porous outer region [1]. Such properties may provide PEO as an ideal underlying layer for the application of solid lubricants which can be entrapped in outside porous and provide reservoirs for the tribological contact lubrication, however the relevant work is scarce. This study investigates the fretting wear behavior and adaptive mechanisms for the PEO produced Al_2O_3 surface of about 11-12 GPa hardness with a top layer of an MoS_2-Sb_2O_3-C chameleon solid lubricating coating, which is named such for its ability to self-adapt tribological contact surface and provide friction and wear reduction in variable humidity [2]. Coupons of AA 6082 alloy were coated by the PEO process and then were over-coated by a burnishing process with a MoS_2-Sb_2O_3-C chameleon coating to prepare such duplex coating combination. The coated surfaces were then subjected to over 10,000 cycles of fretting wear against steel and alumina balls with variable amplitude (0 to 100 micrometers) and loads (10-100 N) in both humid air and in dry nitrogen, including cycled (air/nitrogen) environment conditions. The tests demonstrated low friction coefficients, considerable reduction in critical amplitude for the stick-slip transition, and self-adaptive tribological behavior in the cycled environment tests. Friction coefficients of the order of 0.10 to 0.15 in humid air and 0.06 to 0.09 in dry nitrogen were recorded and linked with the surface self-adjustment from graphite to MoS_2 lubrication, respectively. Raman, SEM and cross-sectional FIB/SEM/EDX analysis of the wear tracks were used to investigate the mechanisms of the adaptation and fretting wear performance. The study demonstrate the effectiveness of the adaptive PEO-Chameleon coating system performance for the fretting wear mitigation in changing environment.

[1] A.L. Yerokhin et al., Surface and Coatings Technology, 122 (1999) 73.

[2] J.S. Zabinski et al., Tribology Letters, 23 (2006) 155.

3:30pm **E1-2-7 Lubricant/Coating Interactions and Their Effect on Tribological Performance: In-situ XAS Analysis of a Dynamic Lubricated Interface**, ***Ardian Morina,*** University of Leeds, UK **INVITED**

The ability to model and predict friction and wear performance in boundary-lubricated conditions is limited by the lack of qualitative and quantitative information on transient tribochemical reactions between the lubricant additives and surfaces. It is these reactions that define tribofilms ' physical and chemical properties, essential for friction and wear performance of industrial boundary lubricated systems, such as valve train, piston ring/liner and pumps. In addition to tribofilm formation, recent work has shown that some of the lubricant additives, such as Molybdenum dialkyl dithiocarbamate (MoDTC), can have a detrimental impact on hydrogenated Diamond-Like-Carbon (DLC) coating durability.

In the current paper, a bespoke tribometer has been designed to be coupled with the Raman spectroscopy and synchrotron X-ray Absorption Spectroscopy for in-situ study of the tribochemical reactions between the lubricant additives and the surface. The focus will be on two typical lubricant additives: Zinc dialkyl dithiophosphate (ZDDP) used as an anti-wear, and MoDTC used as a friction modifier additive. The growth of tribofilms on heat-treated and DLC coated surfaces, in relation to friction and wear performance, will be discussed in detail.

4:10pm **E1-2-9 Friction and Wear Mechanism of MoS_2/C Composite Coatings under Atmospheric Environment**, ***Peiling Ke***, *S Cai, A Wang,* Ningbo Institute of Materials Technology and Engineering, Chinese Academy of Sciences, China

Tribological properties of MoS_2/C coatings with different carbon contents (44.7~84.3 at.%) deposited by magnetron sputtering were systematically investigated under atmospheric environment. During tribological tests, the coating with the least MoS_2 content exhibited the lowest friction coefficient and wear rate, while coating with the most MoS_2 showed the worst performance. To understand friction and wear mechanism, multiple analytical tools such as SEM, EDS, Raman, XPS and TEM were applied to investigate the composition and structure. TEM and SEM characteristics proved that the tribofilm with multilayered structure was formed on the tribo-pair. The C rich layer adhered to the tribo-pair and the top layer was well-ordered MoS_2 tribofilm, and the dominated amorphous MoS_2 was found between the two layers. It suggested that the shear plane was mainly made of well-ordered MoS_2 transfer film, while carbon improved the mechanical properties of the coatings, served as a lubricant and also inhibited the oxidation of MoS_2.

4:30pm **E1-2-10 Adhesion and Mechanical Properties of Ti Films Deposited by DC Magnetron Sputtering**, ***RobertoCarlos Vega-Morón***, *G Rodríguez-Castro,* Instituto Politecnico Nacional, Surface Engineering Group, Mexico*; D Melo-Máximo,* Tecnológico de Monterrey-Campus Estado de México, Mexico*; J Méndez-Méndez,* Instituto Politécnico Nacional, Mexico*; L Melo-Máximo,* Instituto Tecnológico y de Estudios Superiores de Monterrey, Mexico*; J Oseguera,* Tecnológico de Monterrey-Campus Estado de México, Mexico

Titanium (Ti) films were deposited by DC Magnetron Sputtering by changing deposition times and substrate temperature on AISI 316L steel to evaluate different thicknesses and its properties. Cristal orientation was determined by grazing angle X-ray diffraction (XRD). Scanning electronic microscope (SEM) was used to determine the surface composition and deposition characteristics through elemental analysis, and also to measure film thickness. Ellipsometry measurements were performed to compare film thicknesses estimated with SEM. Surface topography was obtained by atomic force microscope (AFM). Using nanoindentation test with a spherical indenter, mechanical properties were estimated. Furthermore, failure mechanisms and critical loads were determined by progressive load scratch tests. Wear behavior was studied through pin-on-disk tests with a 6 mm-diameter WC ball. SEM and optical profilometry were used to examine wear tracks; wear rates and coefficient of friction were analyzed.

4:50pm **E1-2-11 Tribology of New Surface Modifications for Cold Rolling Mill Rolls**, ***Henara Costa***, Universidade Federal do Rio Grande, Brazil*; J Gonçalves Jr., J de Mello,* Universidade Federal de Uberlandia, Brazil **INVITED**

The present work analyzed the tribological behavior of coatings/surface modifications traditionally used in cold rolling mill rolls and new coatings/surface modificationswith potential to replace the carcinogenic hard chrome. The study started with identification of wear mechanisms occurring in real coldrollingmill rolls. Due the high cost and dimensions of the rolls, thereplication technique was used. Replicas were obtained from 4 different rolling millBrazilian companies before and after a normal rolling campaign. Initial sliding tests were conducted using spherical and cylindrical counter bodies in order to verifywhichtribological conditions allowed to reproduce the wear mechanisms found in the replicas. These tests indicated the use of reciprocating sliding tests with cylindrical counter bodies (line contact), normal load of 100 N, and test times of and 1 h and 5 h. Different surface modifications were carried out on samples produced from a fragment of a rolling mill roll. The specimens were heat treated and ground on both sides. After, some specimens were surface textured by electrical discharge texturing (EDT). For both groups (ground and EDT), subsequent treatments of chromium plating, electroless NiP coating and plasma nitriding were carried out. The results of the reciprocating tests showed that specimens with electroless NiP coating presented the lowest friction coefficients, while plasma nitrided specimens showed the highest. In general, previous surface texturing before the coating/surface modification increased the wear of the counter bodies. Oneexception was for EDT with subsequent electroless NiP coating, which presented the lowest counter bodies wear rate. The samples withelectroless NiP coating promoted a tribolayer consisting of Nickel, Phosphorus and Oxygen on both the specimens and thecounter bodies, which was apparently responsible for the reduction of friction coefficient and wear rate. The increase of the test time reduced the wear rate of the samples, apparently due the stability of the tribolayers formed, except for the nitrided samples. For the textured specimens, NiP coating showed the best performance in maintaining the surface topography of the specimens after the sliding tests

Tribology and Mechanical Behavior of Coatings and Engineered Surfaces
Room Royal Palm 4-6 - Session E1-3
Friction, Wear, Lubrication Effects, and Modeling

Moderators: Albano Cavaleiro, University of Coimbra, Carsten Gachot, Vienna University of Technology, Nazlim Bagcivan, Schaeffler Technologies GmbH & Co. KG, Germany

8:00am **E1-3-1 A Study on the Tribological Behavior of the AISI 316L Steel Exposed to Boriding to Reduce its Friction Coefficient and Enhance its Wear Resistance**, ***Enrique Hernández- Sánchez,*** Instituto politécnico Nacional-UPIBI, Mexico*; J Velazqez,* Instituto Politécnico Nacional-ESIQIE, Mexico*; A Chino-Ulloa,* Instituto politécnico Nacional-UPIBI, Mexico*; I Torres-Avila,* Instituto Politecnico Nacional-UPIBI, Mexico*; J Castrejón-Flores,* Instituto politécnico Nacional-UPIBI, Mexico*; H Herrera-Hernández,* Universidad Autónoma del Estado de Mexico, Mexico

The AISI 316L stainless steel is well known by its high resistance to corrosion and its low response to the human fluids. Those are the main reasons that make it considered as a steel alloy suitable to medical applications. This study is on the application of the boriding process to an AISI 316L steel to evaluate its effect on the tribological behavior of it. The boride layers were achieved by applying the powder pack boriding process. The treatment time was set in 2, 4 and 6 h at temperatures of 900, 950 and 1000 °C. The morphology of the layer was evidenced by Scanning Electron Microscopy and nature of the boride layers was analyzed by X Ray Diffraction. The mechanical properties were evaluated by both instrumented nanoindentation and Vickers micro hardness test. The tribological behavior of the layers was evaluated by means of a sand/rubber apparatus by following the limits of the ASTM G-65 standard. The friction coefficient of the borided layers was estimated by means of the tribological pin-on-disk tests. The results showed a clear influence of the experimental parameters on the thickness of the boride layers. Also the mechanical properties were affected by the parameters of treatment especially by the temperature. The wear resistance of the layers tended to increase as the layer thickness increased according to the treatment conditions. However, wear mechanisms such as adhesion and micro-fatigue were mainly observed in the samples exposed to 6 h and 1000 °C. Finally, the friction coefficient was diminished from values of 0.7 for the as-received material to 0.2 for the borided samples.

8:20am **E1-3-2 Immersion Time-affected Tribocorrosion Behavior of Cr/GLC Multilayer Coating in Artificial Seawater**, ***Lei Li,*** *L Liu, P Ke, A Wang,* Chinese Academy of Sciences, China

A chromium/graphite-like carbon (Cr/GLC) multilayer coating was deposited onto 316L stainless steel by direct current (DC) magnetron sputtering technique to enhance the tribocorrosion resistance of the substrate in marine environment. Taking into account intermittent use of some friction components accompanying long-time immersion in seawater, the influence of immersion time varying from 4 h to 48 h on the tribocorrosion behavior of Cr/GLC multilayer coating was studied in artificial seawater by a reciprocating ball-on-plate tribometer and an electrochemical workstation. The results of tribocorrosion tests after different time of immersion showed that the friction coefficient of Cr/GLC multilayer coating continuously increased from 0.070 to 0.085 with prolonged immersion time, while wear rate firstly decreased and reached its minim value of 5.20×10^{-7} mm^3/Nm at 12 h then gradually increase as the immersion time was in the range of 12 h to 48 h. Eectrochemical impedance spectroscopy (EIS) analysis, before and after the tribocorrosion test, clearly demonstrated that the corrosion resistance of Cr/GLC multilayer coating varied with immersion time, which is associated with the competitive effects of corrosion products blocking the micropores and contact stress promoting microcrack initiation and propagation. Such varying corrosion resistance led to time-affected tribocorrosion behavior of Cr/GLC multilayer coating, thus, more attention should be paid to the performance variation of protective coatings for tribocorrosion applications after long-term exposure to corrosive environments.

8:40am **E1-3-3 A Comparison of the Galling Wear Behaviour of PVD Cr and Electroplated Hard Cr Thin Films**, ***Jaimie Daure,*** *P Shipway, G McCartney,* The University of Nottingham, UK

PVD chromium coatings exhibit good mechanical properties and are a possible replacement to electroplated hard chromium (EPHC) in various applications. Electroplated Cr is widely used but there is need to find an alternative to EPHC due to environmental legislation. One possibility is to use PVD Cr thin films as a replacement. However, insufficient information exists on the behaviour of PVD Cr. Therefore, the aim of the study was to compare the behaviour of two PVD Cr films deposited by different processes and compare with EPHC. Galling testing was selected as it is a useful method for testing the wear resistance and adhesion of a coating under high stresses.

Two PVD chromium coatings of approximately 8 µm thickness were investigated and compared to electroplated chromium of ~9.8 µm. The two PVD coatings were deposited by magnetron sputtering and electron beam physical vapour deposition (EBPVD). Coatings were deposited on 316 stainless steel substrates. Samples were characterised by SEM, XRD, EDX and profilometry. Mechanical testing consisted of nanohardness (ISO 14577-4), scratch testing (ASTM C1624-05) and galling testing (ASTM G98-02).

The nanohardness and surface roughness of the two PVD coatings were similar (around 5 GPa and 300 nm Ra respectively), the values were roughly double for the EPHC. All coatings exhibited similar scratch behaviour. Galling tests revealed that the EBPVD provided no improvement in galling resistance compared to self-mated stainless steel (22 MPa). The magnetron sputtered PVD and 9.8 µm EPHC failed at almost six times the stress of the EBPVD (125 Mpa).

Top surface SEM revealed the two chromium PVD coatings to have distinctly different microstructures. XRD revealed the EBPVD chromium coating had a strong preferred orientation in the {2 0 0} planes, whereas the magnetron sputtered chromium coating had preferred orientations in the {1 1 0} and {2 1 1} planes. The electroplated chromium revealed low intensities of crystalline peaks suggesting a lack of crystalline order or an orientation effect.

Overall, the hardness and surface roughness of the coatings appeared not to be a dominant factor in galling resistance. The crystal orientation of the PVD chromium coatings appears to play a large role in galling resistance. As BCC materials contain slip planes of type {1 1 0} in the <1 1 1> direction, the coating with a preferred orientation in the {1 1 0} planes is expected to provide better wear resistance due to the slip planes parallel to the surface. In the presentation, mechanisms for galling will be discussed and related to future development of thin Cr coatings for galling resistance.

9:00am **E1-3-4 Microstructural Evolution of Cold-sprayed Copper Coating during Reciprocating Sliding Wear**, ***Yinyin Zhang,*** McGill University, Canada*; C Greiner,* Karlsruhe Institute of Technology (KIT), Institute for Applied Materials (IAM), Germany*; D Chern, R Chromik,* McGill University, Canada

Cold-sprayed Cu is characterized by various deformed microstructures including nano- and submicron-grains due to dynamic recrystallization, coarse grains containing dislocation forests, as well as deformation bands, deformation twins. When those microstructures were subjected to sliding wear loading, they turned into equiaxed micrometric recrystallized grains below the topmost tribofilm that consisted of nanocrystalline grains. The present work focuses on microstructural evolution mechanisms of the subsurface layer, i.e. grain boundary migration of the nano- and submicron-grains under tribological loading.

First, sliding wear tests were carried out on the as-sprayed and annealed Cu coatings and the subsurface microstructure before and after sliding were observed and analyzed by electron backscattered diffraction (EBSD) maps. This allows, firstly, to compare the difference between thermal-driven and stress-driven grain growth, and secondly, to explore the role of internal energy on microstructural evolution during sliding, given that annealing at different temperatures (i.e. 200 °C, 300 °C, 400 °C, and 500 °C) was to decrease the stored energy gradually. It was found that thickness of the sliding-induced grain growth layer deceased with annealing temperature and disappeared in the 400 °C-annealed specimen. Microstructural features (e.g. grain size, geometrically necessary dislocation density, texture, etc.) of this layer with increase in annealing temperature were discussed based on the EBSD maps. Nanoindentation was used to measure the hardness of the subsurface microstructures. The hardness profiles of the subsurface indicated lower hardness of the grain-growth layer compared to the initial microstructure and this layer became thinner with increase in annealing temperature and eventually disappeared at 400 °C. These results suggest a stored energy criterion probably existed, above which the grain boundary migration occurred under tribological loading.

9:20am **E1-3-5 Scratch Adhesion Resistance of Nickel Boride Layers on Inconel 718 Superalloy**, *I Campos-Silva, **Alan Contla-Pacheco**, A Ruiz-Rios, J Martínez-Trinidad, G Rodríguez-Castro, A Meneses-Amador, W Wong-Angel,* Instituto Politecnico Nacional, Surface Engineering Group, Mexico

New results about the scratch adhesion resistance of nickel boride layer on Inconel 718 superalloy were estimated in the present study. The nickel boride layer was developed on the surface of Inconel 718 superalloy by means of the powder-pack boriding process conducted at 1173 K with 2, 4, and 6 h of exposure. The microstructure of the nickel boride layer was analyzed from optical microscopy, X-ray diffraction and energy dispersive spectroscopy (EDS) in order to verify the presence of Ni_4B_3, Ni_2B, Ni_3B and the distribution of alloying elements along the depth of the layer; the thicknesses of the nickel boride layer + diffusion zone were established between 23 to 40 micrometres for all the set of boriding conditions. Furthermore, and before the scratch tests, indentation properties of the nickel boride layers such as hardness, Young's modulus, plastic deformation resistance, and the distribution of residual stresses were estimated using Berkovich nanoidentation tests applying a constant load (50 mN) across the diffusion layers. The scratch tests were performed over the surface of the nickel boride layer-substrate systems using a Rockwell-C diamond indenter with a continuously increasing normal force from 1 to 80 N, whereas the behavior of the coefficient of friction and the residual depth as a function of the scratch length were monitored during the tests. For the determination of the critical loads, the combination of acoustic emission signal with microscopic observations of the worn tracks were used; the critical loads were estimated at which the layer cracks (cohesive failure) or is detached (adhesive failure) and they explained according to the mechanical properties of the nickel boride layer-substrate system. For all the set of experimental conditions, the presence of different failure mechanisms over the worn tracks was detected, while the results showed that the critical loads increase with increasing nickel boride layer thickness.

9:40am **E1-3-6 Comparison of Surface Treatments for Adhesive Force Measurements Between Magnetron Sputtered TiW Thin Films and Alumina Substrates**, *B Atabay, **Elif Apaydin**,* Aselsan Inc., Turkey

This work evaluates the adhesion characteristics of magnetron sputtered Titanium Tungsten (TiW) thin films to surface treated polycrystalline alumina (Al_2O_3) substrates. The experimental relationship has been established in the same region of substrates between four different sets of surface treatments of alumina (Al_2O_3) and TiW thin film adhesion. A progressive load scratch test for adhesion was performed between two surfaces by applying a gradually incremented vertical load and measuring the opposing horizontal force. The 132 N of adhesive force can be obtained by suitable surface pre-treatment of alumina. Results were evaluated by optical microscopy, SEM and surface scratch measurement techniques.

10:00am **E1-3-7 Influence Of Microstructure on Wear of Boroaluminized-Hot-Work Tool Steels**, ***Undrakh Mishigdorzhiyn***, *N Ulakhanov,* East Siberia State University of Technology and Management, Russian Federation*; Y Chen, H Liang,* Texas A&M University, USA

This research investigates a possibility in improving wear resistance in hot-worked toolsteels by means of high-temperature boroaluminizing in treatment pastes. Boroaluminizing was conducted in sodium fluoride (as an activator), boron carbide and aluminum (BC:Al=4:1) in a furnace. The treatment was administered for 4 hours at 1100°. The microstructure, microhardness, and phase compositions of the boroaluminized layers were evaluated and their effects on wear were studied. Results indicated that high-temperature boroaluminizing improves wear resistance of hot worked tool steels.

Tribology and Mechanical Behavior of Coatings and Engineered Surfaces

Room Royal Palm 4-6 - Session E3

Tribology of Coatings for Automotive and Aerospace Applications

Moderators: Sebastien Guimond, Oerlikon Balzers, Oerlikon Surface Solutions AG, Nicolas Argibay, Sandia National Laboratories, Christian Greiner, Karlsruhe Institute of Technology (KIT), Institute for Applied Materials (IAM)

2:10pm **E3-3 Cladding Tribaloy T400 on Steel Substrates using a High Power Nd:YAG Laser**, ***Wei Ya***, *B Pathiraj, D Matthews,* University of Twente, Netherlands*; M Bright,* Tata Steel, Netherlands*; S Melzer,* Tata Steel Research & Development, Netherlands

Tribaloy T-400 is a Cobalt and Molybdenum based alloy, which has been developed for the resistance to high temperature wear, galling and corrosion. Its hardness is provided by a hard inter-metallic Laves phase, dispersed in a tough matrix of eutectic or solid solution. However cracking during processing limits its application such as hard facing using laser surface cladding/coating, especially when a Nd:YAG laser is applied as is commonly available in industry. The primary aim of this work is accomplished by laser cladding crack-free Tribaloy T-400 layers using a high power Nd:YAG laser. The optimal process conditions of cladding crack free Tribaloy T-400 coating on different steel substrates (st355J2 steel plates and AISI 316 steel bar) were obtained. The dilution effects on the hardness of cladded Tribaloy T-400 coating are investigated. Microstructures of the clad layer produced with optimal process parameters with and without preheating substrate were analysed by using Scanning Electron Microscope (SEM). The chemical compositions of different phases in the clad were analysed by using Energy Dispersive X-ray Spectroscopy (EDX). The resulting Austenite, Ferrite and Laves phases present in the clad were analysed by using X-ray Diffraction (XRD). The residual stress in the clads were evaluated by using hole drilling and XRD techniques. The correlation between the process conditions and resulting microstructures are discussed to provide guidelines for further up-scaling of laser clad crack-free Tribaloy T-400.

2:30pm **E3-4 Tribological Properties of HVOF-Sprayed WCCoCr Coatings for Applying to Sliding Rings of Mechanical Seals**, ***Aleksander Iwaniak***, Silesian University of Technology, Poland*; G Wieclaw,* Certech Sp. z o.o., Poland*; L Norymberczyk,* ANGA Sp. z o.o., Poland

The critical elements of mechanical seals are sliding rings. In almost all applications constructions, at least one of these rings is made of tungsten carbide or silicon carbide, usually in the form of a solid ceramic. The paper presents the results of research on the development of a new generation of sliding rings with a metallic core and a working face covered with coating using HVOF thermal spray technology with the use of WCCoCr ultra fine powder. The metallic core of the ring provides very good mechanical properties, including impact resistance, while the heat-sprayed carbide coating allows for wear resistance comparable to that made of solid ceramic. The tests were conducted with the "ring on ring" method for time 400 hours. The specimens "ring" were made of various materials: carbon-graphite material and a tungsten carbide. The condition of the top layer of the specimens was determined after the friction tests (3D profilometry, SEM). Laboratory tests of the new generation of slip rings and preliminary tests in industrial conditions have shown that the developed solution is characterized by high durability and has high application potential.

Financial support by The National Centre for Research and Development (NCBiR) in Warsaw, Poland - Project No INNOTECHK2/IN2/2/181798/NCBR/13 is gratefully acknowledge.

2:50pm **E3-5 The Effects of Relative Humidity on Fretting Corrosion Behaviors of Silver-plated Electrical Contacts**, ***Florent Pompanon***, *S Fouvry,* LTDS, CNRS UMR 5513, Ecole Centrale de Lyon, Ecully, France*; O Alquier,* PSA, Vélizy – Villacoublay, France

During the last decades, the use of connectors in electrical devices for automotive has increased significantly. This raise in the number of electrical and electronic devices on board cars has led to a growing number of breakdowns. Indeed, this connectors need to keep a low and stable electrical contact resistance (ECR) otherwise micro-interruptions of signal may occur. Due to their work environment (car engine) they are subjected to vibrations inducing fretting in the contact.

Fretting occurs at the interface of materials in contact and refers to small oscillatory motion between the two surfaces. This phenomenon induces wear and the formation of oxide debris layer (third body) in the contact area increasing the electrical contact resistance.

The damaging effect on electrical contacts has been widely studied, a lot of studies have been conducted on non-noble materials (such as tin-plated contact) and noble materials (gold-plated and silver-plated contact) to determine the mechanisms of electrical contact damage and predict the electrical contact resistance endurance (ECR endurance)[1], [2].

The aim of this study is to consider the effect of the relative humidity rate on fretting behavior of silver-plated electrical contact. A climatic generator is used to work in a range of relative humidity (RH) from 10 % to 90 %.

The results show that the relative humidity rate impact the electrical contact resistance endurance. The number of cycles to reach the electrical failure (Nc) of the contact increase substantially, and especially when the relative humidity is above 50 %. Two behaviors can be highlighted, a threshold RH_{th}=50% as been established marking the transition between the two regimes :

1. When RH < 50% :

- A rather constant evolution is observed, the ECR endurance remains stable.

- The third body layer is formed of pulverulent oxides displaying a low current conductivity . They are easilly ejected from the interface. This implies a high wear rate and a low ECR endurance.

1. When RH > 50% :

- The ECR endurance strongly increases : the larger the relative humidity the large the ECR endurance (linear increase).

- The formation of oxide – hydrate is activated. The third body is more compacted and adherente to the interface (uneasy third body ejection), the wear rate is decreased. Moreover, hydrates displays a lower coefficient of friction and high current conductivity extending the ECR endurance.

These hypotheses are discussed regarding the mechanical, chemical and electrical characterization of the debris layer.

References:

1. S.Fouvry, P. Jedrzejczyk, P. Chalandon, Wear 271, 2011, 1524-1534

2. J.Laporte, O. Perinnet, S. Fouvry, Wear 330-331, 2015, 170-181

3:10pm **E3-6 Evaluation of Solid Particle Erosion Resistant Coatings for Gas Turbine Engine Applications**, ***Qi Yang***, National Research Council of Canada, Canada

Aircrafts, when operating in a sandy environment , can experience severe erosion damage to gas turbine engine components, such as compressor blades, vanes and impellor blisks/wheels, due to sand particle ingestion. As erosion progresses, a significant amount of material removal not only leads to significant aerodynamic losses, but results in the structural weakening of blades as well. Applying erosion resistant coatings on airfoil surface has been proven effective in extending the serviceable life of engine components. In the course of coating development, the adequate erosion testing techniques have to be applied in order to identify potential candidates economically and under representing conditions. From this perspective, various erosion testing techniques are first reviewed for their pros and cons. Then, proper testing protocols and evaluation methods of erosion resistance are elaborated based on testing results of TiN coating and another proprietary erosion resistant coating using gas jet apparatus. Taking the consideration of aerodynamic factors to erosion performance, wind tunnel sand erosion testing is also performed on the coatings. The results of gas jet and wind tunnel erosion testing are compared and discussed. Furthermore, several technical concerns related to erosion resistant coatings are deliberated.

3:30pm **E3-7 Influence of Sliding Induced Defects on the Frictional Properties of Molybdenum Disulfide (MoS_2) and Graphene**, ***Zaixiu Yang***, *S Bhowmick, G Sun,* University of Windsor, Canada*; F Sen,* Argonne National Laboratory, USA*; A Alpas,* University of Windsor, Canada

2D-layered structures MoS_2 and graphene show low coefficient of friction (COF) during initial sliding contact (running-in period), but their long-term tribological performance depend on the moisture in the testing

environment. Sliding induced defects contribute differently to frictional properties of MoS_2 and graphene under humid environments. Micro-Raman spectroscopy indicated that sliding induced structural defects in graphene, but graphene showed a low friction under humidity while a higher friction in dry N_2. Using van der Waals interaction incorporated Density Functional Theory (DFT) calculations, it was found that dissociative adsorption of water molecules at a mono-vacancy site of the graphene led to the increase in the graphene layer spacing and reduced interlayer adhesion energy, and thus contributed to the reduced COF. Meanwhile, the MoS_2 tested under humid environment showed a high friction whereas a low friction in dry N_2 was observed. Results of DFT calculations suggest the possibility of formation of Mo-O-Mo by the dissociated water molecules at triple vacancy site, resulting in the increase in COF as will be discussed in the conference.

3:50pm **E3-8 Analysis of Tribo-mechanical Behavior of a Low Temperature Plasma Nitrided Austenitic 316L Stainless Steel**, *J Oseguera,* ITESM-CEM, Mexico*; R Meza,* TEROMOINNOVA, Finland*;* ***Fernando Santiago****,* ITESM-CEM, Mexico

Microstructure of AISI 316L nitrided steel, treated by a weak ionized plasma process, using N2,H2 and Ar gas mixtures, was analyzed. Active nitrogen in the plasma that correlates to the diffusion of nitrogen in the steel is identified by optical emission spectroscopy. The kinetic evolution of expanded austenite was identified by treatments developed at low temperatures bellow the eutectoid transformation temperature. Phases obtained on surface were identified by XRD, from cross sectional views obtained by optical microscopy the thickness of expanded austenite was measured. Vickers hardness profile from surface was measured. Analyses of tribo-mechanical behavior of steel in a pin-on-disk system, for nitrided and non-nitrided steels samples, were used to contrast friction coefficients. The response of the tribo-mechanical system was interpreted through the structural characterization of the steel.

4:10pm **E3-9 Tribological Systems Solutions for Gas Turbine Engines**, ***Pantcho Stoyanov****, A Wusatowska-Sarnek, T Kasprow,* Pratt & Whitney, USA **INVITED**

The advancement of durable gas turbine engine components depends heavily on the development of high-performance materials, which can withstand extreme environmental and contact conditions (e.g. large temperature ranges, high contact pressures, and continuous impingement of abrasive particles, all of which degrade the physical properties). In particular, due to the large number of complex contacting and moving mechanical assemblies in the engine, the lifetime of certain structures is limited by the tribological performance of the employed materials and coatings. This talk will provide an overview of tribological solutions employed in several sections of gas turbine engines. After a general review of aircraft engine tribology, the talk will focus on coatings used for clearance control (i.e. abradable air seals) as well as tribological materials used to minimize fretting type of wear. More specifically, a study will be presented on the influence of self-lubricating hexagonal boron nitride (hBN) on the erosion and abradability of Ni-based abradable coatings. Subsequently, a series of studies on the friction and wear behavior of Ni-based and Co-based superalloys at elevated temperatures will be presented. Emphasis will be placed on the correlation between the third body formation process (e.g. oxide layer formation, transferfilms) and the tribological behavior of the superalloys. This talk will conclude with a discussion of the needs for tribological coating solutions in gas turbine engines.

4:50pm **E3-11 The Friction and Wear Performance of DLC Coatings Deposited on Plasma Nitrided AISI 4140 Steel by Magnetron Sputtering under Air and Vacuum Conditions**, ***Halim Kovacı,*** Atatürk University, Turkey*; O Baran,* Erzincan University, Turkey*; A Yetim,* Erzurum Teknik University, Turkey*; Y Bozkurt, L Kara,* Erzincan University, Turkey*; A Çelik,* Atatürk University, Turkey

Diamond-like-carbon (DLC) coatings with high hardness and low friction coefficient exhibit excellent tribological performance under air and vacuum conditions. However, adhesion and cold welding problems in vacuum conditions lead to increase friction coefficient values. These negative effects can be eliminated by different methods such as forming interlayers between coating and substrate or ion treatment of the substrate. In this work, DLC coatings were deposited on untreated and plasma nitrided (at 400 °C, 500 °C and 600 °C for 1h and 4h) AISI 4140 steel substrates by magnetron sputtering technique. The effects of plasma nitriding treatment on the friction and wear properties of DLC coatings under air and vacuum conditions were investigated. The structural and mechanical properties of DLC films were examined by XRD, SEM, and microhardness tester, respectively. The friction and wear properties were determined by a tribotester under air and vacuum conditions. The microhardness of samples increased after surface treatments and the highest value was obtained from the sample plasma nitrided at 600 °C for 4h plus DLC coated sample. The wear resistance of samples increased with increasing plasma nitriding time and temperature. Also, it was observed that the samples tested under vacuum condition showed better wear resistance than the samples tested under ambient air. Furthermore, increasing plasma nitriding time and temperature improved the wear performance of the material regardless of the test environment.

Tribology and Mechanical Behavior of Coatings and Engineered Surfaces
Room Grand Hall - Session EP
Symposium E Poster Session

EP-2 Effect of Power on Soft Magnetic and Tribological Properties of Fe-Co based Coating by Laser Cladding, ***Xiaoshan Yang***, *X Cui, G Jin, J Liu,* Harbin Engineering University, China

Fe-Co based coatings which have a broad application in improving the surface properties have excellent wear resistance and soft magnetic properties. In this study, (FeCoNi) Si B Cu Mo coatings with high saturation magnetization and good tribological properties were prepared using lasers cladding.The different process parameters effect on the microstructure, mechanical properties of the cladding layer wereinvestigated. It was found that laser power P, scanning speed V and laser remelting technology which had a significant effect on the magnetic properties and tribological properties. The optimum process parameters were determined and the coatings showed bcc dominated microstructures. The average microhardness and friction coefficient of the coatingsreached 1032 $HV_{0.5}$ and 0.291 respectively. With the decrease of grain size, wear resistance and microhardness of coatings improved significantly. In addition, the coatings exhibited higher saturation magnetization (212emu/g), comparable magnetic properties to those of their conventionally processed counterparts.

Acknowledgements

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EP-3 Tribological Behavior of the FeB Phase in Boron Coating Formed on an AISI L6 Steel using Ball On Disc with Dry Conditions, ***Daniel Sanchez Huerta***, CBI, Universidad Autónoma Metropolitana unidad Azcapotzalco, Mexico*; I Hilerio Cruz,* Universidad Autonoma Metropolitana unidad Azcapotzalco, Mexico*; N Lopez Perrusquia,* Universidad Politecnica Del Valle De Mexico, Mexico*; E García Bustos,* Catedras CONACYT, Mexico, México*; M Doñu Ruiz,* Universidad Politecnica del Valle de Mexico, Mexico*; M Flores Martinez,* Universidad de Guadalajara, CUCEI, Mexico

The coefficient of friction (CoF) and wear behavior in a boron coating was studied at different temperatures of 1173, 1223 and 1273 K, with residence times of 0.5, 2 and 3 h for each temperature. The boron coating was obtained by powdered box technique on AISI L6 steel (DIN 1.2714). The roughness of each treatment was studied by roughness profile (2D) measurements, obtained an increase in R_a between 0.352 (1173 K with 0.5 h) to 0.965 µm (1273 K with 3 h) for the FeB phase. The reciprocating tribometer tests were studied at normal loads of 7 and 10 N with humidity and temperature controlled in dry conditions. For the FeB phase, we obtained CoF values from 0.49 to 0.64 at 36 m of displacement. The topography of the wear traces, were analyzed by optical profilometer (3D). The wear in the FeB phase obtained in the sliding tests, suggested a resistance wear of the treatment at high thicknesses; in the Fe_2B phase the wear was not obtained, because the thickness wear is around from 0.44 to 0.98 µm. The wear behavior are two bodies with an adhesive behavior in the FeB phase.

EP-4 Tribocorrosion Behavior of Boronized AISI 4140 Steel, ***Steffen Aichholz***, *R Torres, M Meruvia, P Soares,* PUCPR, Brazil

In this study, AISI 4140 steel was boronized using solid state thermochemical boriding technique, producing a monophasic iron boride (Fe_2B) layer with a mean thickness of 49,5 µm. The microstructural and chemical characteristics were evaluated by Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS) and X-ray Diffraction (XRD). To investigate the tribocorrosion behavior samples were immersed in 3,5% NaCl solution subjected to a pin-on-disk wear test and connected to a potentiostat monitoring the open circuit potential (OCP).

The results obtained show that boronized samples have an improved tribological behavior compared to unboronized samples and the evaluation of the OCP curve indicates the formation of a passivation layer during de wear test. These results can be explained by the high hardness and chemical inertness of iron boride layer.

EP-5 Influence of Sputter Power Ratio on Microstructure, Mechanical and Tribological Properties of Ti-B-C Coatings Deposited onto AISI M2 Steel, ***Elbert Contreras***, *M Gómez,* Universidad de Antioquia, Colombia

Despite being a ceramic material widely known for its high hardness, chemical resistance and high thermal stability; TiB_2 coatings using in industrial conditions has been really limited due to the high residual stresses associated with the reduction of adhesion. Recently, several investigations have shown that the incorporation of carbon within TiB_2 coatings reduces the residual stresses, increasing the adhesion and becoming Ti-B-C coatings in excellent candidates for applications at high temperatures. In this research, Ti-B-C coatings were deposited onto M2 steel substrates by DC UBMS. Prior to the coatings deposition a plasma cleaning process was carried out both substrates and targets with a 40 sccm flow of Ar and a pressure of 3 Pa for 0.5 h. For the deposition, two targets of TiB_2 (99.9%) and graphite (99.9%) opposite each other was used. The power density of TiB_2 was fixed to 2.4 W/cm^2 and four different power ratio W_{TiB2}/W_C was made in order to varied the carbon content in the Ti-B-C coatings. Microstructural analysis by X-ray diffraction (XRD) showed h-TiB_2 crystal structure with preferential orientation (001), increasing the carbon content, the intensity of (001) peak decrease becoming the crystalline coatings in amorphous coatings. Using AFM technique, an increase in roughness and grain size was observed with higher carbon content, except for coatings with ~20%. SEM images revealed columnar, dense and homogenous structure for all coatings. A progressive decreasing in hardness and Young modulus was observed with the increasing in carbon content, hardness up to 27 GPa was reached with lower carbon content and a decreasing under 5 GPa with the higher carbon content. The Young modulus exhibited the same featured. Tribological properties of the coatings were investigated using Pin-on-disk, all coatings showed COF higher tan AISI H13 substrate, only the coatings with 36% of carbon showed COF over 0.30. respecting to the wear rate it was keep almost constant for all Ti-B-C coatings.

EP-6 Structural and Mechanical Properties of W-doped HfO_2 Thin Films, *A Uribe, M Garcia, R Chintalapalle,* ***Cristian Orozco***, University of Texas at El Paso, USA

Hafnium oxide (HfO_2) is a high temperature ceramic with interesting mechanical properties. HfO2 is distinguished by its wide band gap (~5.7 eV), high dielectric constant (20-25), and high chemical stability. In an effort to tune and enhance the structural and mechanical properties, this work was focused on doping tungsten (W) into HfO_2 thin films. While the doping effects of other metals have been studied, there is a lack of knowledge on the effects of W doping into HfO_2. On the other hand, refractory metal incorporated dielectrics are expected to meet the functional requirements of high-temperature device applications. W-doped HfO_2 thin films were deposited on silicon and quartz substrates via radio frequency magnetron sputtering. The W content was varied by changing the sputtering power from 0-100 W. The films were characterized by performing x-ray diffraction (XRD), nanoindentation, scanning electron microscopy (SEM), and contact angle measurements. Nanoindentation studies revealed increases in hardness and elasticity with increasing W content. XRD displayed the predominant (-111) peak of monoclinic HfO_2 and revealed amorphization induced by increasing W concentration. SEM data also showed gradual amorphization in the samples with increasing W-content. Finally, contact angle measurements showed all the samples were hydrophilic. Based on the experimental data, a structural-mechanical-property relationship is established.

EP-7 Tribological Studies on Self-Lubricating (Cr,Al)N/MoS_x Coatings at Elevated Temperature, *K Bobzin, T Brögelmann,* ***Nathan Kruppe***, *D Hoffmann,* Surface Engineering Institute - RWTH Aachen University, Germany*; F Klocke, P Mattfeld, D Trauth, R Hild,* Laboratory for Machine Tools and Production - RWTH Aachen University, Germany

Due to the high material utilization and the associated resource and energy efficiency, production processes from the field of cold forging of steel are of great importance. At present, environmentally harmful lubricants have to be used to ensure process stability as well as low wear and friction. Due to environmental, economic and legislative aspects, there is an increased research potential to reduce or to completely substitute lubricants. To achieve the goal of lubricant free dry cold forging of steel, physical vapor deposition (PVD) coatings with self-lubricating properties are applied on forming tools. Promising for this application are PVD coatings on the basis of a (Cr,Al)N hard phase with embedded MoS_x which simultaneously meet the requirements of high wear resistance and friction reduction. Furthermore, a sufficient compound adhesion is needed to withstand high

contact stresses up to σ = 3,000 MPa during cold forging of steel. In addition to the mechanical stresses, a thermal load up to T = 250 °C occurs during cold forging. In the presented work, three self-lubricating coatings (Cr,Al)N/MoS_x deposited with varying bias voltage were investigated. The hybrid PVD technology, consisting of direct current and high power pulse magnetron sputtering dcMS/HPPMS, was used for the coating deposition in an industrial coating unit. Two different steels AISI D2 (X155CrMoV12, 1.2379) and AISI M2 (HS6–5–3C, 1.3343) were used as substrate materials. The influences of the substrate material and heat treatments at T = 250 °C on mechanical, compound adhesion and tribological properties were investigated. The samples were analyzed with respect to the universal hardness HU and modulus of indentation E_{IT} using nanoindentation. To determine the interfacial adhesion of the compound coating/substrate, Rockwell indentation and scratch tests were carried out dependent on the heat-treatment. For the analysis of the tribological behavior, AISI 5115 (16MnCr5, DIN 1.7131) and AISI 4140 (42CrMo4, DIN 1.7225) were used as counterpart materials, since these are widely used as forming material. As closed tribometer, a Pin-on-Disc (PoD) was used at varying temperatures. In this case, the coated substrate is continuously loaded with the worn pin on the same track. As open tribometer, a newly developed Pin-on-Cylinder (PoC) was used. The particular feature of the PoC is that the pins (AISI D2) are coated and pressed by means of a defined feed along a rotating cylinder (AISI 5115 and AISI 4140). Thus, the coated pin is permanently in contact with an unworn surface of the cylinder. The contact region was studied by Raman spectroscopy before and after the tribological tests.

EP-8 Role of Carbon Nanotubes in Reducing Friction between Steel/Steel Contacts, ***Zaixiu Yang**, S Bhowmick,* University of Windsor, Canada*; F Sen,* Argonne National Laboratory, USA*; A Alpas,* University of Windsor, Canada

Carbon materials such as graphene and carbon nanotubes (CNT) are increasingly used to reduce the friction. By performing sliding tests on steel against steel immersed in ethanol solutions with 0.005 mg/ml of CNT added, the coefficient of friction (COF) was reduced from 0.27 (in absolute ethanol) to 0.16. Once the CNTs were oxidized, the partially unzipped CNT showed reduced agglomeration in ethanol. Moreover, the partially unzipped CNT in ethanol led to the elimination of the running-in stage of friction and more stable COF curves. Examination of the worn surfaces indicated that the deposition of CNT to the steel surfaces, which was confirmed by the micro-Raman spectroscopy, was responsible for the reduced COF. Density Functional Theory simulation results were used to reveal the effect of the partially unzipped structure of CNT on the improved friction properties as will be discussed in the presentation.

EP-11 Microstructure Change, Element Diffusion and Tribological Properties of Chromium Oxide from RT to 1000 °C, ***Huidi Zhou**, N He, X Liu,* Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, China

The demands of reusable solid lubricants with continuous lubricating properties from room temperature (RT) to 1000 °C or even higher temperatures become increasingly urgent with the development of high-tech industries, especially that of aerospace industry. In our work, chromium oxide was investigated as a primary coating because it possesses high melting point and excellent thermal stability. The chromium oxide coatings were deposited on Ni-based high-temperature alloy substrates through an arc ion plating system and then annealed at 1000 °C in air for 2 h. The effects of annealing on the structure and tribological properties in wide temperature range of Cr_2O_3 coating were researched in detail. The phase change and elements diffusion behavior of Cr_2O_3 coating annealed at 1000 °C were also discussed. Main results and conclusions are as-followed:

Ti and Cr atoms in the Inconel 718 alloy matrix diffuse to the coating surface and react with oxygen in atmosphere, thereby generating the mesh-like heave structure that endues the annealed coating an excellent self-lubricating behavior in wide temperature range. The friction coefficients are all lower than 0.3, and the wear rates maintain at a magnitude of 10^{-7} mm^3/Nm from RT to 1000 °C. Particularly, the excellent self-lubricating performance could be maintained even in five-thermal-cycles test. Moreover, while the mesh-like heave structure damaged by friction, Ti and Cr atoms in the matrix tend to diffuse to the damaged locations and react with oxygen in air. This phenomenon results in the reproduction of composite-phase heave structure of Cr_2O_3 and $Cr_2Ti_4O_{17}$ at the damaged locations. Consequently, the self-supplement ability of the mesh-like heave structure could prolong the wear life in wide temperature range.

EP-12 Sliding Wear Behaviour of Infiltrated Self-lubricating Polymer Matrix Composites Studied by in-situ Tribometry, ***Yinyin Zhang**,* McGill University, Canada*; R Schulz,* Hydro-Québec Research Institute (IREQ), Canada*; R Chromik,* McGill University, Canada

The hydropower industry routinely makes use of thermally sprayed coatings and solid lubricating polymer coatings in their operations to enhance the lifetime of their infrastructure, especially those components subjected to sliding wear. Often these two coating systems are used independent of one another but in this study, we examine the feasibility of a duplex coating where a self-lubricating polymer composite was infiltrated into a porous SS316 substrate. This resulted in coatings consisting of a top layer made of a self-lubricating polymer composite and a bottom layer made of a polymer composite infiltrated into a porous metallic structure. This paper presents the tribological behaviour of the polymer composite and the transition when wearing reaches the infiltrated metallic porous substrate.

Addition of graphite (Gr) to a thermoset bismaleimide (BMI) significantly reduced coefficient of friction and wear rate. An in-situ tribometer that uses a transparent sapphire hemisphere as counterfaces permits microscopic observation and video recording of the sliding contact. Interfacial sliding dynamics such as material transfer, formation of transferfilms and wear debris were examined and therefore wear mechanisms of BMI + Gr and BMI + Gr + SS316 were observed. A stable transfer film that was rich in Gr was formed when sliding on the top layer of BMI + Gr, while participation of metallic material in the sliding contact contributed to deposition of transfer film onto wear tracks and transfer film reformation. Raman spectroscopy was used to detect formation of Gr-containing tribofilms and transferfilms in the running-in and steady-state regimes. X-ray photoelectron spectroscopy (XPS) testing on the wear tracks and transferfilms revealed possible tribochemical reactions such as polymer decomposition and interaction with Gr and/or SS316 induced by sliding wear. Mechanical properties of the as-received polymer composites and worn materials were also investigated using nanoindentation.

EP-13 Sputtered B-C-W-Coatings: Composition – Properties – Stability, ***Heidrun Klostermann**,* Fraunhofer FEP, Germany*; J Poetschke,* Fraunhofer IKTS, Germany*; O Zywitzki,* Fraunhofer FEP, Germany

Coatings in the material system B-C-W have been synthesized by pulsed magnetron co-sputtering of a boron carbide and a tungsten target. The bipolar co-sputtering process enables fine tuning of the composition through variation of sputtering pulse times. In the range of composition from pure boron carbide to 70 at-% tungsten content, the hardness of the coatings as determined by nanoindentation changes only little, typically in the range of 24 GPa to 28 GPa. However, in contrast to completely amorphous pure boron carbide, the x-ray diffraction pattern of B-C-W coatings exhibit broad peaks, indicative of extremely small crystalline domains that cannot clearly be attributed to a single phase for tungsten content below 50 at-%. Corresponding to a smooth transition in structure, scanning electron microscopy investigation reveals very homogeneous coatings with extremely smooth surfaces, even at a thickness of several microns. Consistently, EDX mapping reveals a very homogeneous element distribution. Thermal stability has been tested up to a temperature of 600°C in vacuum and in air. While no degradation is observed in vacuum, the oxidation rate in air depends on the tungsten content of the coatings.

In contrast to many other hard coatings, the stress level of the B-C-W layers is very low and the material seems to be rather resistant to crack propagation, as far as scratch test analysis can reveal. The coatings have been tested on cutting tools for cemented carbide cutting, where they abrade rather rapidly. However, the combination of properties still makes them interesting as hard and tough coatings.

EP-14 Comparison of Tribological and Electrochemical Properties of Titanium Oxided Films Produced on Cp-Ti by Sol-Gel and Silar Methods, *O Çomaklı,* Erzincan University, Turkey*; M Yazıcı,* Erzurum Technical University, Turkey*; **Halim Kovacı**,* Atatürk University, Turkey*; T Yetim,* Erzurum Technical University, Turkey*; A Yetim,* Erzurum Teknik University, Turkey*; A Çelik,* Atatürk University, Turkey

Cp-Ti is commonly used in different applications because of its good structural properties but its low mechanical and tribological properties restricts its usage areas. Therefore, different surface modifications based on TiO_2 formation on material surface are applied to enhance its surface properties. In this study, TiO_2 films were produced on Cp-Ti substrates by sol-gel and successive ionic layer adsorption and reaction (SILAR) methods in order to compare the performance of different coating methods. The structural, morphological and mechanical features of TiO_2 films were

investigated by XRD, SEM and microhardness tester. The tribological properties of films were characterized using a pin-on-disc tribotester. The electrochemical behavior of TiO_2 films were determined by potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) analyses. The structural and mechanical analyses showed sol-gel method caused the formation of more stable and hard film structures with good adhesion in comparison to SILAR method. For that reason, TiO_2 films produced by sol–gel method exhibited better wear and corrosion resistance than the films produced by SILAR.

EP-18 Mechanical and Tribological Properties of W–C–N Films Using Unbalanced Magnetron Sputtering Assisted by Linear Ion Source, ***Hyundong Kim,*** *S Heo, E An, I Park,* Korea Institute of Industrial Technology (KITECH), Republic of Korea

Ternary W–C–N films were deposited on 2618 aluminum alloy and 304 stainless steel substrates by a hybrid deposition process combining d.c. unbalanced magnetron sputtering and linear ion source system using pure tungsten targets in an $N_2/(N_2+Ar)$ gas mixture for diesel engine pistons applications. The influence of the carbon content and deposition conditions, i.e. substrate bias voltage and temperature, on the microstructure, mechanical properties and tribological properties of the W–C–N films was systematically investigated in this study. The microstructure for the films was characterized by X-ray diffractometer (XRD), scanning electron microscope (SEM), X-ray photoelectron spectroscope (XPS), and high-resolution transmission electron microscope (HRTEM). Nano-indentaion was conducted to assess the nanohardness and Young's modulus of the W–C–N films. Wear resistance and tribological behavior of these films also were evaluated using a micro-tribometer with wear debris analyses and coefficient of frictions. Moreover, morphological analysis with surface roughness was calculated by using atomic force microscope (AFM).

EP-19 The Influence of Feedstock Powders on Microstructure and Tribological Properties of WC-Co-Cr HVAF Coatings, *K Szymański, G Moskal, D Niemiec,* ***Aleksander Iwaniak,*** *J Wieczorek,* Silesian University of Technology, Poland

The basic goal of presented investigations is comparison of feedstock powders of WC-Co-Cr type in area of its chemical and phase compositions as well as morphological parameters, size distribution and shape. Those parameters have direct transfer on technological properties of powders such as followability etc., what is essential form high velocity spraying process point of view. In this part of article the X-ray and EDS analysis of powders were made, additionally scanning electron microscopy observation was carried out too. The size distribution was analysed by laser diffraction method. Obtained data gives the possibility to comparison of powders from different sources from technological properties point of view.

The second part of paper related with microstructural characterization of coatings deposited by high velocity air fuel (HVAF) method. The standard procedure for those kind of powders was used. The gun of Kermetico of K5 type was used. The range of investigations included characterization of coatings top surface with descriptions of roughness, as well as the stress state was analysed by X-ray method. Phase analysis was made after deposition and obtained data were compared to initial phase composition of powders as well as theirs chemical compositions. In area of mechanical properties basic parameters such as hardness and fracture toughness were measured and analysed. The final part of investigations is related with characterization of tribological properties of coatings with the same chemical and phases constituent buy deposited from different feedstock materials

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EP-20 Microstructure and Mechanical Properties of CuSn10 Alloy Coating Manufactured by Cold Spraying, ***Weihuang Liu,*** *J Cao, Z Yin, H Li, G Gao,* Shanghai Jiao Tong University, China

In order to achieve compact and low oxidation of Cu-10wt.% Sn (CuSn10) alloy coating, the cold spraying is used to deposit a coating on the bearing material substrate. The coating is analyzed by its morphology, hardness, metallographic, porosity and finite element analysis. Results show the mechanical performances of CuSn10 coating that are excellent. The microstructure of coating is compact and its hardness is appropriate for the requirement of embeddability and machinability. The sizes of grains of CuSn10 are uniform. There are no obviously grain boundary cracks. The porosity ratio is 0.07 %. Stress and deformation show that coating has excellent ability of reducing vibration and absorbing energy. With coating protection, results of fatigue life show that life is improved 173.32 %. The microstructure and mechanical properties of CuSn10 are outstanding that the cold spraying coating is very suitable for bearings.

EP-22 Scratch Induced Thin Film Buckling for Quantitative Adhesion Measurements, *A Kleinbichler,* KAI – Kompetenzzentrum Automobil- und Industrieelektronik GmbH, Austria*; J Zechner,* KAI - Kompetenzzentrum Automobil- und Industrieelektronik GmbH, Austria*;* ***Megan Cordill,*** Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Austria

The adhesion of thin films is one of the most important factors for the reliability of microelectronic devices and the semiconductor industry requires quantitative testing methods to effectively compare these interfaces. Several techniques have been developed over the last decades such as four point bending, budge testing, micro cantilever tests, spontaneous buckling and scratch induced delamination. For compressively stressed films on rigid substrates the scratching can cause buckling failure ideal to be used to determine the adhesion quantitatively by modeling the delaminations according to the Huchinson and Suo model. Two different sample systems, a tungsten-titanium film on a silicate glass and a silicon nitride film on a silicate glass with a tungsten-titanium overlayer, have been tested using a ramp load method in the range of 100-500mN. This study demonstrates that the scratch induced delaminations resulted in parallel and spontaneous buckles. The parallel buckles developed directly beside the scratch trace and were the result of fracture events at the interface and deformation left by the passing indenter tip. The spontaneous buckles, on the other hand, originated from the parallel buckles and propagated according to the stress distribution in the film forming telephone cord delaminations. Using the geometric dimensions of the induced buckles the adhesion energies of the interfaces were quantitatively calculated. It was shown that the adhesion energy of the tungsten-titanium film increased with annealing time from 2.7 J/m² for the as-deposited to 4.7 J/m² for the 2 hour annealed film at 400°C. The adhesion of the silicon nitride film was determined to be 1.4 J/m² and was in good agreement with previous nanoindentation experiments. The results illustrated that the scratch test can be utilized for quantitative adhesion testing of thin films in cases where other methods are not applicable or sample preparation would change the nature of the interface and suggest that scratch induced delamination is a valuable addition to established adhesion measurement techniques.

EP-23 Study of the Mechanisms of Built-up Edge Formation during Machining of Super Duplex Stainless Steel, ***Yassmin Seid Ahmed,*** *G Fox-Rabinovich, B Bose, D Covelli, J Paiva, G Dosbaeva, S Veldhuis,* McMaster University, Canada

Tool wear is a very important factor determining tool life and surface quality of a machined workpiece surface. So, it is necessary to minimize tool wear to maximize tool life and to optimize the manufacturing performance. Different mechanisms can cause the tool wear in a specific machining process, adhesion onto the cutting tool frequently observed during machining of stainless steels, generate damage on the tool rake face. Adhesion of the workpiece material causing built-up edge (BUE) formation tends to promote tool chipping, since BUE is never completely stable, but it periodically breaks off leading to tool failure. The phenomenon of BUE formation can lead to poor machined surface texture and accelerated tool wear, subsequently leading to increased manufacturing costs. An experimental study was undertaken in order to study the formation and the cutting mechanism of the BUE. In this paper, the mechanisms that trigger the formation of BUE during the machining of super duplex stainless steel alloys—Grade UNS S32750 with PVD AlTiN/TiSiN coated carbide tool has been investigated. The process parameters were chosen so that the BUE formation was provoked. The BUE formation and tool wear was evaluated throughout the cutting tests using an Alicona Infinite Focus microscope and a scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (EDS). Tribo-film formation on the worn rake surface of the tool was analyzed using X-ray Photoelectron Spectroscopy (XPS). Comprehensive characterization of the BUE mechanisms of PVD AlTiN/TiSiN coated vs. uncoated cutting tool wear was performed using SEM, electron backscatter diffraction (EBSD) and nanohardness tester. This paper investigates the deformation mechanisms and plastic behavior of austenite and ferrite phases in BUE cross section. Nonohardrness distribution and EBSD phase mapping of BUE cross section samples revealed a built-up of austenite bands are collected at the tool-chip interface and ferrite bands collected on the top. Moreover, SEM

images of BUE cross section samples show micro-cracks which act as BUE initiators were identified in this region of austenite.

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