Tribology and Mechanical Behavior of Coatings and Engineered Surfaces

Room San Diego - Session E2-1

Mechanical Properties and Adhesion

Moderators: Gerhard Dehm, Max-Planck Institut für Eisenforschung, Etienne Bousser, The University of Manchester, Fan-Bean Wu, National United University, Taiwan

1:50pm **E2-1-2 Cross-sectional Investigation of Microstructure and Mechanical Properties of Graded Ti(N,B) Coatings***, **Michael Tkadletz**, N Schalk, C Mitterer, C Hofer, J Keckes,* Montanuniversität Leoben, Austria*; M Deluca,* Materials Center Leoben Forschung GmbH, Austria*; M Pohler, C Czettl,* CERATIZIT Austria GmbH, Austria

Chemical vapor deposited (CVD) TiB_2 coatings are typically grown on a thin TiN base-layer to prevent diffusion of B into the substrate. While CVD TiN coatings exhibit a rather large grain size in the µm range, tensile residual stress and a relatively low hardness, the TiB_2 coatings are characterized by a nanocrystalline microstructure, high compressive residual stress and high hardness. In order to alleviate the resulting sharp transition at the interface of these two layers, an additional Ti(N,B) layer with B content gradually increasing from pure TiN to pure TiB_2 was introduced. Subsequently, the coating was investigated using scanning- as well as transmission electron microscopy, cross–sectional synchrotron X-ray nanodiffraction and cross–sectional dynamic nanoindentation and modulus mapping techniques. A grain size gradually decreasing from the µm to nm range, a significant change of residual stress from 0.5 to -2 GPa and an increase in hardness from ~18 to ~45 GPa with increasing B content could be observed across the coating thickness. With increasing B content, a transition from the face centered cubic to the hexagonal structure was also found. The formed compounds and their three dimensional arrangement was investigated by combinatorial use of Raman and X-ray photoelectron spectroscopy as well as atom probe tomography. The obtained results provide the basis for an evolutionary design of TiN/TiB_2 and Ti(B,N) coatings with optimized B content.

2:10pm **E2-1-3 Nanocrystalline Pt-Au MEMS Electrical Switches***, **Nicolas Argibay**, M Dugger, D Adams, C Nordquist, A Grine, M Henry, P Lu,* Sandia National Laboratories, USA

Microelectromechanical systems (MEMS) relays have orders of magnitude higher figure of merit compared to semiconductor devices, along with lower power consumption and insertion loss. Unfortunately, MEMS switches have not penetrated high volume applications partly due to contact adhesion, contamination, high cycle switch life and microstructural evolution leading to performance drift.

Recent work has shown that it is possible to achieve extraordinarily stable nanocrystallinity in some binary metal alloys via solute segregation in the as-deposited state, even at high temperatures. One of these alloys, Pt-Au, may provide solutions to several challenges preventing greater adoption of MEMS switches. As-deposited nanocrystalline Pt grains are stabilized by the presence of Au at grain boundaries, presenting the possibility of reduced creep and relaxation in the electrical contacts and structural elements of a MEMS switch. Sputtered Pt-Au was used to construct MEMS switches, and initial results suggest performance improvements relative to baseline switches made from Au. MEMS switch performance and associated material evolution mechanisms will be discussed.

2:30pm **E2-1-4 Thin Film Metallic Glass: Novel Coating Providing High Toughness and Low Friction***, **Chia-Chi Yu**, J Chu,* National Taiwan University of Science and Technology, Taiwan*; Y Shen,* University of New Mexico, USA

The amorphous nature of thin film metallic glasses (TFMGs) provides outstanding mechanical properties, including high strength, large elastic limits, and excellent corrosion and wear resistance. The grain boundary-free structure of TFMGs produces an exceptionally smooth surface and low surface free energy, resulting in high hydrophobicity and a low coefficient of friction.

In this study, magnetron sputtering was used in the deposition of Zr-based TFMG coatings with the aim of enhancing the bending and fatigue properties of bulk metallic glasses (BMGs). A TFMG coating was shown to increase the plastic strain of BMG by 9.2%, without sacrificing its extraordinary strength. This was also shown to increase the fatigue endurance-limit of BMG by ~33%, from 300 MPa for bare BMG to 400 MPa for TFMG-coated BMG. The results of transmission electron microscopy and nanoindentation testing revealed that TFMGs are able to withstand enormous shear strain without fracturing. Used as a coating on syringe needles, the low coefficient of friction of TFMG (∼0.05) reduced the insertion forces by ∼66% and retraction forces by ∼72%, when tested on polyurethane rubber blocks.

2:50pm **E2-1-5 Driving Force for the Texture Transformation of Thin Metal Films***, E Ellis,* Cornell University, USA*; M Chmielus,* University of Pittsburgh, USA*; S Baker,* Cornell University, USA*; Y Cheng, P Liu, **Ming-Tzer Lin**,* National Chung Hsing University, Taiwan **INVITED**

The texture transformation has been attributed to a competition between strain energy and interface energy. In FCC materials, the (111) orientation has the lowest interface energy, while the (100) orientation has the lowest biaxial modulus. Thus, at a given strain, the interface energy per unit volume decreases as the inverse of the film thickness, while the strain energy per unit volume is constant with film thickness. However, recent studies have questioned the role of both stresses and interface energies in this texture transformation. Where stresses are known with certainty, they have been shown to be insufficient to produce the texture transition, and the transitions seem to occur in films of similar thickness regardless of the interface conditions. We simulated the driving forces using a first principle density functional theory for the orientation selection mechanisms and investigated the transformation by using a bulge test apparatus to induce different stresses in thin Ag films under identical annealing conditions. *In situ* synchrotron XRD measurements show the change in texture during annealing, and reveal that applied stresses have no effect on the transformation. Stress analysis shows that differences in driving forces for texture transformation due to applied bulge pressure were significant (≈200 kJ/m^3), suggesting that a different, much larger driving force must be responsible. Reduction in defect energy has been proposed as an alternative. However, vacancy and dislocation densities must be exceptionally high to significantly exceed the strain energy and do not provide obvious orientation selection mechanisms. Nanotwins in reported densities are shown to provide greater driving force (≈ 1000 kJ/m^3) and may account for orientation selection. The large difference between the calculated strain and defect energies and the driving force for grain growth (21,100 kJ/m^3) casts doubt on the applicability of a thermodynamic model of texture transformation.

3:30pm **E2-1-7 Strength and Strain Hardening Behavior of Particle Strengthened Coherent Cu/Ni Multilayer Films***, **Rachel Schoeppner**, M Polyakov, G Mohanty, J Michler,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland

There are many techniques used to increase the strength of a material depending on the application and desired results. Two such techniques include deposition of nanolaminate structures and the addition of hard particles to act as dislocation barriers. Particles have been known to act as barriers to dislocation motion as well as Frank-Read dislocation sources, which both increases the strength and strain-hardening ability of the material. This type of strengthening has traditionally only been applied to bulk alloys and oxide dispersion strengthened materials with no investigations focusing on the combined affect of particles distributed at the interface of nanoscale multilayer films. Some molecular dynamic simulations focusing on steps and imperfections specifically at the interface of Cu/Nb multilayer films have shown additional strengthening can occur as the step size of the kink increases; however, the presence of these interfacial defects can also result in an initial decrease of the yield strength as they act as dislocation sources, initiating plasticity sooner than in films with pristine interfaces, which is similar to the concept of Frank-Read sources in traditional particle strengthened films. The extent of the interaction of hard W nanoparticles deposited at the usually coherent interface of Cu/Ni multilayer films has been investigated through both nanoindentation and micropillar compression to determine both the strengthening and strain-hardening affect of these particle enforced interfaces. Initial investigations have shown an overall increase in the nanoindentation hardness of these films by a maximum of 1 GPa when compared to CuNi multilayers without particles at the interface. However, as the particle density increases the amount of strengthening is actually shown to decrease in the range of volume fractions investigated here. Further microstructural and mechanical characterizations have also been conducted to more fully explain this phenomenon.

3:50pm **E2-1-8 Indentation Induced Deformation and Damage in Metal-Ceramic Multilayer Coatings***, **Yu-Lin Shen**, R Jamison,* University of New Mexico, USA

Metal-ceramic multilayer composite coatings are an exciting subset of materials with many promising attributes. This presentation highlights our recent studies on mechanical characterization of such coatings using the nanoindentation technique. We focus on aluminum (Al)/silicon carbide (SiC) nanolayers, which serves as a model system for investigating the constraining effect due to the highly mismatched mechanical properties of the constituents. How this structural heterogeneity can affect the indentation behavior is a current a subject of active research. The development of complex deformation patterns underneath the indentation, dictated by the structural heterogeneity, can lead to various forms of local damage. Our studies focus on the employment of numerical finite element modeling to corroborate with experimental observations as well as to extract meaningful constitutive properties. Special attention is given to the analyses of (i) plastic deformation in the metal layers, (ii) cyclic indentation response and composite modulus measurement and (iii) indentation-induced delamination.

4:10pm **E2-1-9 Influence of Various Interlayers on Mechanical Properties of CrAlN Coatings on Tungsten Carbide Substrate***, **HoeKun Kim**, J La, M Song, S Lee, Y Hong,* Korea Aerospace University, Republic of Korea

Among many ternary nitride protective coatings, the CrAlN coatings have been paid much attention to the cutting tool's coating applications due to their excellent properties such as high hardness, low surface roughness, and excellent thermal stability. It was reported that the interlayer with the median hardness to elastic modulus ratio (H/E ratio) between the value of the coating and the substrate improved the wear resistance of the coating. In this work, various interlayers such as CrN, CrZrN, CrN/CrZrSiN were synthesized between the CrAlN coating and the tungsten carbide substrate to improve mechanical properties of the coatings. All the coatings were produced by an unbalanced magnetron sputtering system on the WC-6 wt.% Co substrate, and total thickness was controlled to be 3 μm. The microstructure, hardness and elastic modulus, and friction coefficient were evaluated by field-emission scanning electron microscopy (FE-SEM), nano-indentation, and ball-on-disc type wear tester, respectively. All the coatings were annealed at temperatures from 600 to 1000˚C in furnace for 30 min, and the hardness values were investigated using nano-indentation.

The hardness and elastic modulus of all the CrAlN coatings were not affected significantly by type of the interlayer, and they were measured to be in the ranges of 35.5 to 36.2 GPa and 424.3 to 429.2 GPa, respectively. However, wear test showed that the CrAlN coatings with the CrN and CrN/CrZrN interlayer exhibited improved friction coefficient of 0.34 compared to the CrAlN coating with the CrZrN interlayer (COF 0.41), and the wear rate and width of those coatings showed lower values. These improved wear properties could be attributed to the H/E ratio of the interlayer between the CrAlN coating and the WC substrate. In view of the coating structure, there exists a gradual decrease in the H/E ratio from the CrAlN coating (H/E, 0.089), to the CrZrSiN interlayer (H/E, 0.083) and CrN interlayer (H/E, 0.076), and the WC substrate (H/E, 0.040). The CrZrSiN and CrN interlayers induced a smooth transition of the stress effectively under loading conditions during the wear test, and this led improved wear resistance of the CrAlN coating. During the thermal stability tests, the hardness of the CrAlN coating with the CrN/CrZrSiN coating was maintained up to 1000˚C due to excellent oxidation resistance of the CrZrSiN layer consist of the amorphous SiN phase .

4:30pm **E2-1-10 Numerical Investigation of Damage and Fracture in Hard Nano-coating Layers using Cohesive Zone Modeling***, **Shahed Rezaei**, S Wulfinghoff, S Reese,* RWTH Aachen University, Germany

Coating layers are usually applied on different manufacturing tools in, e.g., plastic extruder in order to increase their lifetime and to improve the surface properties of the final parts. New coating deposition techniques such as high-power impulse magnetron sputtering (HPPMS) can provide more parameters to control the coating mechanical properties, therefore they result in producing coating layers with better performance and perhaps higher damage resistance. In order to be able to compare the effect of different parameters on damage behaviour of the coatings, a cohesive zone (CZ) element model has been applied. The fracture modes are divided into an intergranular fracture inside the coating and delamination between the coating layer and the substrate. The developed numerical model allows predicting the damage initiation and propagation within various types of coating systems in different setups such as nanoindentation. Numerical studies of nanoindentation tests show that the intergranular cohesive tractions, residual stresses, elastic and plastic properties and the grain morphology of the coating layers are the most effective parameters in order to produce stronger coatings.

Tribology and Mechanical Behavior of Coatings and Engineered Surfaces
Room San Diego - Session E2-2

Mechanical Properties and Adhesion

Moderators: Gerhard Dehm, Max-Planck Institut für Eisenforschung, Etienne Bousser, The University of Manchester, Fan-Bean Wu, National United University, Taiwan

8:00am **E2-2-1 Study of Bauschinger Effect in Ni Thin Metallic Films Submitted to Cyclic Deformation***, **Pierre-Olivier Renault**, W He, P Godard, E Le Bourhis, P Goudeau,* Universite de Poitiers, France

The lifetime of flexible electronic devices is strongly dependent on their mechanical performance as they are submitted to complex thermo-mechanical cyclic loadings during service. As an elementary substructure inside such devices, metallic thin films are often supported by a polymer substrate.

A substantial amount of experimental work has shown that the plastic response of a thin metal film can be very different from that of its bulk counterpart. The yield stress, flow stress and hardening rate of thin films depend on film thickness. During cyclic deformation of a material, the plastic deformation in one direction can affect the plastic response in reverse direction; one consequence is the decrease of the yield strength of a metal when the direction of strain is changed, i.e. a reduced elastic limit at reversal straining. Such Bauschinger effects have been reported on different metallic thin films in the last ten years.

In this communication, an experimental method using uniaxial tensile testing is used to study the Bauschinger effect in thin metal films deposited on pre-stretched polyimide substrate. Thanks to our new pre-stretch setup based on previous work [1], the metallic thin films can be deformed alternatively from tension to compression within a strain domain of a few % (depending on the elastic range of the polymer substrate). The elastic intra-granular strain of polycrystalline thin films and true strain of substrates are measured in situ during tensile-compressive loading by X-ray diffraction (XRD) and digital image correlation (DIC) techniques. A complete strain transfer through the interface is observed in the elasto-plastic regime as the interface is strong enough thanks to the thin film elaboration PVD technique (namely ion beam sputtering) [2]. From lattice strain-true strain curves, the mechanical response of thin film/substrate set is analyzed in view of the complete loading history.

[1] Renault P.-O., Faurie D., Le Bourhis E., Geandier G., Drouet M., Thiaudiere D., Goudeau P., “Deposition of ultra-thin gold film on in situ loaded polymeric substrate for compression tests”, Materials Letters **73**, 99-102 (2012).

[2] Geandier G., Renault P.-O., Le Bourhis E., Goudeau Ph., Faurie D., Le Bourlot C., Djemia Ph., Castelnau O., Cherif S. M., Elastic-strain distribution in metallic film-polymer substrate composites, Applied Physics Letters 96, 041905 (2010).

8:20am **E2-2-2 Mo-Re Thin Films for Flexible Display Applications***, F Hauser, T Jörg,* Montanuniversität Leoben, Austria*; M Cordill,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Austria*; R Franz,* Montanuniversität Leoben, Austria*; H Köstenbauer, J Winkler,* Plansee SE, Austria*; **Christian Mitterer**,* Montanuniversität Leoben, Austria

Sputtered Mo films are nowadays widely used as thin films in flat panel display applications, e.g., for gate and source/drain electrodes or signal and data bus-lines. However, due to their brittleness, usability in future flexible displays is limited. In order to overcome this disadvantage, a strategy to improve the ductility of brittle Mo thin films by alloying with Re is explored within this work. A series of Mo-Re thin films with 50 nm thickness were grown on 50 µm thick polyimide substrates by magnetron co-sputter deposition from pure Mo and Re targets. Up to ~25 at.% Re, a solid solution with the body-centered cubic structure of Mo was obtained. In-situ characterization methods were applied to determine the electro-mechanical behavior of the films during deformation. Uniaxial tensile tests were performed under the light microscope to directly observe the fragmentation process and to determine the crack onset strain. In addition, the electro-mechanical response was evaluated by measuring the change in the electrical resistance during straining. After tensile straining, pure Mo thin films exhibited straight through-thickness cracks, which are usually observed for brittle films, while the Mo-Re films showed a wavy crack path, indicating a more ductile behavior. The addition of Re also has a significant effect on the electro-mechanical response of films, where the rise of the resistance indicated a crack onset strain three times higher than for pure Mo. In summary, alloying of Mo thin films with Re is a promising strategy to improve their ductility, which in turn can enable their utilization in flexible displays.

8:40am **E2-2-3 Rate Sensitive and Creep Behavior of Thin Metallic and Oxide Films: on Chip Testing and Activation Volume Analysis***, **Thomas Pardoen**, G Lemoine, H Idrissi,* Université Catholique de Louvain, Belgium*; D Schryvers,* University of Antwerpen, Belgium*; M Ghidelli,* Université Catholique de Louvain, Belgium, Italy*; M Coulombier, R Vayrette, L Delannay,* Université Catholique de Louvain, Belgium*; S Gravier,* Grenoble INP, France*; J Raskin,* Université Catholique de Louvain, Belgium **INVITED**

Creep and viscoplastic deformation mechanisms are generally amplified in thin films compared to bulk systems due to various factors related to the dominance of the free surfaces and/or to the abundance of internal defects and interfaces. A series of experimental investigations on freestanding thin metallic, metallic glass and oxides films using an on chip uniaxial test method will be reviewed, focusing on the creep/relaxation behavior. The generic approach to tackle with thermally activated mechanisms through the activation volume mechanics framework is applied to rationalize the measurements. These measurements are supplemented by in or ex situ transmission electron microscopy analysis and micromechanical models to unravel the origin of the dominant deformation mechanisms in connection with the microstructure. The commonalities and specificities among these systems will be discussed. An important common characteristic is the improved ductility associated to a enhanced rate sensitivity.

9:20am **E2-2-5 Intrinsic Stresses - New Methods to Evaluate Them Using Enhancing Indentation Methods and New Models to Optimize Them***, **Nick Bierwisch**, N Schwarzer,* SIO, Germany

In many d eposition processes intrinsic (or residual) stresses can't be avoided during the coating creation. Mostly because of bias or high deposition temperatures and the mismatch in the coefficients of thermal expansion for the various materials. The intrinsic stresses can have a big influence on the material behavior in contact situations.

In one way they can help fighting against your critical external loads and reducing the created stresses. On the other way they can also weaken the material compound when producing to much stresses in a weaker part of the system. Both sides can also have an effect on the adhesion strength between the different coatings. So gaining knowledge about these intrinsic stresses could help a lot in the field of modeling or simulating your worst case application scenarios.

This talk will show 2 new methods to evaluate the intrinsic stresses using extended indentation measurements and new mathematical models. One method applies a mixed load indentation by adding a lateral load component to the applied normal load. The other new measurement uses a reference probe with known intrinsic stresses. With this new measurement methods and new mathematical models the intrinsic stresses within your material can be evaluated.

The second part of the talk will focus on a new model, which allows you to optimize the intrinsic stress distribution to increase the performance in a given worst case application. Nowadays more and more production processes allow to steer the intrinsic stresses during the coating deposition process. This allows a new degree of freedom in the development process and can speedup the development. You can save a lot time because you don't need to search for new materials which also can improve the performance. So knowing how to build up the intrinsic stresses before the deposition process will speedup the development and optimization process of your new material compound a lot. You can save a lot of prototypes and therefore much development time and costs. This work is part of the EU project IStress [1] and within this project this model was implemented into the software package FilmDoctor [2]. It allows you to define your later application and the software will find an intrinsic stress distribution within your coatings which will decrease the resulting stresses in your worst case contact in your later application.

References:

[1] www.stm.uniroma3.it/iSTRESS

[2] [http://www.siomec.de/FilmDoctor]

9:40am **E2-2-6 Investigation of Buckling Driven Delamination of DLC Coatings for Evaluation of Adhesion Strength***, **Richard Braak**, U May, L Onuseit, G Repphun,* Robert Bosch GmbH, Diesel Systems, Germany*; M Guenther, J Emmerlich,* Robert Bosch GmbH, Germany*; C Schmid, K Durst,* Physical Metallurgy, TU Darmstadt, Germany

Diamond like carbon coatings (DLC) are widely used as wear-resistant coatings, e.g. in the automotive industry for Diesel injection systems. Their exceptional properties, as high hardness and high modulus are the result of the ion bombardment during the plasma coating process. In addition the thin films sustain substantial residual compressive stresses which can be a problem with respect to the adhesion: The in-plane compressive stress can lead to buckling driven delamination. It occurs in different shapes: Circular, straight-sided or worm-like blisters or even big-area delamination. The type of appearance is closely linked to the mode-dependent fracture toughness of the interface.

In the current work, ta-C coatings (tetragonal amorphous carbon) with different kinds of adhesion layer design are investigated. As the mechanical properties of the whole coating system do have influence on the buckling behavior, depth profiles of the layered structures are taken via nanoindentation on a small angle cross section (SACS). The intrinsic stresses are determined with two separate methods: The curvature test and via a focused ion beam (FIB) in combination with digital image correlation (DIC).

In the first part of the paper commonly used indentation and nanoscratch tests are discussed, the latter with constant and increasing load. The created damages are investigated thoroughly via SEM-imaging (Scanning Electron Microscopy). The findings lead to a novel adhesion test which is presented in the second part. A ranking of the adhesion strength of the different adhesion systems can be done with the suggested method. The ranking is used to show the range of application of the common scratch and indentation tests.

10:00am **E2-2-7 Characterization of Thin Films by Nanoindentation: Avoiding Mistakes during the Measurement and Data Analysis***, **Esteban Broitman**,* Engineering Consulting, Sweden

Nowadays, nanoindentation has become a routinely technique for the mechanical characterization of thin films and small-scale volumes. Thanks to the development of friendly analysis software and advances in high sensitive instrumentation, it feels like the measurement and calculation of hardness and elastic modulus can be done automatically by just *"the pushing of one button."* However, the consequences of the easy procedures have led many researchers to publish erroneous data [1].

In this paper, common mistakes in the measurement and data analysis during the nanoindentation of thin films will be critically reviewed, and the possible ways to correct them will be discussed: 1) the misuse of the 10%-thickness *"rule of tomb"* to avoid the effect of the substrates; 2) the lack of thermal drift correction in long-term duration experiments; 3) the wrong data conversion from Vickers microindentation to Berkovich nanoindentation; 4) the ignorance of pile-up effects; 5) the misinterpretation of indentation size effects at low penetrations; 6) the wrong determination of tip area functions; 7) the lack of load frame compliance correction during the characterization of very hard coatings; 8) the confusion of thermal drift with creep and viscoelastic effects; 9) the misinterpretation of pop-ins and pop-outs; 10) the preconceptions about a direct relationship between hardness and tip penetration; 11) the preconceptions about a direct relationship between elastic modulus and hardness; 12) the lack of considering surface roughness influence; 13) the possible change of surface mechanical properties during sample preparation; 14) the use of dirty or damaged tips; 15) the natural differences in the results when using spherical, cube-corner, or Berkovich indenters; 16) ignoring the influence of indentation loading rate; 17) the interpretation of elastic recovery in very elastic or very plastic films; 18) the confusion of load-penetration nanoindentation curves with stress-strain compression diagrams; 19) the difference in the results between load-controlled and depth-controlled indentations; and 20) the lack of knowledge about possible work/strain hardening effects or phase transformations during indentation.

The origins of the aforementioned mistakes will be elucidated from the lack of understanding on contacts mechanics theory, the limits and validation of Oliver and Pharr's method, and preconceptions transmitted from generation to generation of nanoindentater users. At the whole, it will be stressed that it is not enough to know *"how to push the button"* in order to measure the nanoscale mechanical properties of thin films.

[1] E. Broitman, Tribology Letters 65 (2017) 23.

10:20am **E2-2-8 Plasma Electrolytic Oxidation Coatings on AZ31 Magnesium Alloys with Si_3N_4 Nanoparticle Additives***, **YiYuan Lin**, J Lee, C Tseng,* Ming Chi University of Technology, Taiwan*; B Lou,* Chang Gung University, Taiwan

The magnesium AZ31 alloys have been used in a wide range of lightweight applications such as aerospace, automotive and personal computers due to its unique properties. However, high chemical reactivity, poor corrosion and wear resistance limit their widespread uses in many fields. The plasma electrolytic oxidation (PEO) process can produce protective oxide layer on the magnesium alloy to improve its mechanical property, wear resistance and corrosion resistance. In this work, the silicon nitride (Si_3N_4) nano particles were added into the electrolyte of PEO treatment on AZ31 alloy to improve the mechanical and anticorrosion properties of oxide coating. Surface and cross-sectional structure of the oxide layers was studied by scanning electron microscope (SEM). Energy dispersive spectrophotometry (EDS), X-ray diffraction (XRD) techniques were employed to determine the phase structure and chemical composition of the layers. The adhesion and mechanical properties of coating were analysis by scratch test, pin-on-disk wear test and hardness test, respectively. Potentiodynamic polarization tests were employed to investigate the electrochemical corrosion behavior of PEO treated AZ31 alloy. Effects of Si_3N_4 addition concentration on the microstructure, mechanical and anticorrosion properties were further discussed in this work.

10:40am **E2-2-9 Fractures, Wrinkles and Buckles in Brittle Multi-layers on Flexible Substrate***, **Davy Dalmas**,* Laboratoire de Tribologie et Dynamique des Système (LTDS), Ecole Centrale de Lyon, France*; I Ben Cheikh, G Parry, R Estevez,* SIMaP – Univ. Grenoble Alpes, CNRS, SIMaP, France

Polymer film coated with stacks of thin layers (metal, oxides or organic) are more and more used in many industrial applications such as flexible and opto microelectronics (screens, OLED, Photovoltaic applications...). Maintaining the mechanical stability of these coated systems requires both to control the cohesion of the coating and its adhesion to the substrate for various mechanical loadings. As the mechanical behaviour of these systems is highly dependent on both the properties of the substrate (modulus, thermal expansion coefficient, glass transition temperature ...), and of the coating (deposition conditions, the residual stresses,…), it is essential to develop dedicated characterization methods and idealized model experiments to understand the mechanical stability of those thin films deposited on flexible substrates.

In this Study, fracture propagation of TiO_2 and Ag layers of various thicknesses coated on a PET substrate is investigated during simple traction tests. During those tests, two phenomena can be observed. Firstly, at low strain, channel crack starts to appear in the coating. We show that the crack density undergoes a transition from a statistic failure distribution classically observed for brittle material and well-described by Weibull distribution to a deterministic sequence of failures set by the elastic mismatch between the film and the substrate. At high strain, the crack density saturates more rapidly than expected (i.e. for distance between two consecutive cracks one order of magnitude higher that he film thickness). Secondly, we observed a second transition from wrinkling to buckling of the coating in the transverse direction to Poisson effect. We show that the transition between those two phenomena is driven by the composition of the coating.

Finally, we propose a two-dimensional model of a film bonded to an elastic substrate to describe the evolution of crack density with the applied strain in the case of large elastic mismatch between the film and the substrate. We then extend this model to an elasto-plastic one in order to account for the plasticity in the substrate. This plasticity is experimentally evidenced by in-situ AFM imaging and numerical validation by FEM that both show the localisation of strain in the substrate during fracture process. We propose that this localisation is responsible of the early saturation of crack density observed at high strain.

11:00am **E2-2-10 Combined XPS and Adhesion Studies of Metal - Polymer Interfaces for Space Applications***, **Barbara Putz**,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Monanuniversität Leoben, Austria*; G Milassin, Y Butenko, C Semprimoschnig,* European Space Research and Technology Centre (ESTEC), The Netherlands*; M Cordill,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Monanuniversität Leoben, Austria

Good adhesion of metal-polymer interfaces is crucial for the reliability of a vast number of high-tech and everyday applications, such as multilayer insulation for satellites as well as flexible and rigid microelectronic devices. Of special interest for space applications is how stable these interfaces are chemically and mechanically with respect to the extreme thermal cycling the devices undergo in operation. In low earth orbit a spacecraft typically encounters 6000 thermal cycles of +/- 100°C during one year in operation. In this study, mechanical adhesion measurements are combined with X-ray photoelectron spectroscopy (XPS) and transmission electron microscopy (TEM) in order to relate the interface strength to the interface chemistry and structure for the Aluminium-Polyimide (Al-PI) system. This material system is used as a multilayer insulator for satellites currently in orbit. The interfacial adhesion energy was measured using tensile induced delamination before and after a thermal cycling treatment of +/- 150°C up to 200 thermal cycles. In order to assess the chemistry of the interface, an 180° peel test was used to provide access to the metal side and the polymer side of the interface. Peeling allows the interface of interest to be evaluated without any additional etching or sputtering steps that would alter the interface chemistry. XPS survey scans and relevant high resolution core levels were recorded on both sides of the peeled interfaces to identify and understand relevant interfacial bonding and to distinguish between adhesive failure of the interface and cohesive failure in the substrate. TEM cross-sections were used to examine the interface structure as a function of thermal cycling and related to the mechanical adhesion measurements. It was determined that the Al-PI system initially has very good metal-polymer adhesion which does not degrade due to the thermal loads caused by the sun during orbit. The combination of mechanical adhesion measurements, structure and chemistry evaluation of the interfaces allows for an improved understanding of how thermal treatments can influence interfacial behaviour between metals and polymers. This new knowledge will help improve design and reliability of the materials used in space applications and can also provide vital information for flexible and rigid microelectronics used on earth.

11:20am **E2-2-11 Mapping Adhesion Energy of Tungsten Based Barrier Layers with Scratch Induced Buckling***, **Andreas Kleinbichler**, J Zechner,* KAI - Kompetenzzentrum Automobil- und Industrieelektronik GmbH, Austria*; M Cordill,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Austria

Diffusion barrier layers provide microelectronic devices with chemical and mechanical stability, thus ensuring the reliability during service. The barrier layer prevents the contamination of the conductive metallization due to Si diffusion in a certain temperature range while also acting as an adhesion layer to the underlying substrate providing mechanical stability. However, the interface adhesion may change due to temperature gradients present during sputter deposition and result in a radius dependent adhesion across the wafer. Tungsten-titanium (WTi) alloys have been demonstrated to be very important barrier materials in copper based microelectronic devices as they exhibit thermal stability up to 800°C and good adhesion behavior to silicon oxide substrates. The adhesion of WTi to Borophosphosilicate glass (BPSG) is of special interest in modern metal–oxide–semiconductor field-effect transistors (MOSFET) since the strength of this kind interface is usually weak. A reliable way of assessing the adhesion quantitatively is required to properly map the adhesion across the wafer surface and for the different temperatures the wafer is exposed to during the production process and later during its lifetime. The adhesion of a 300nm WTi film with an 800nm BPSG substrate on top of a 700µm Si wafer has been investigated using scratch testing. The scratches induce buckles in the film which are used to quantitatively calculate the film/substrate-adhesion and can easily be performed as a function of the position over the wafer. From the different positions a map of the adhesion energy of the WTi-BPSG interface can be constructed. The adhesion map will help identify areas on the wafer that might have been influenced by the thermal gradient present during production.

Tribology and Mechanical Behavior of Coatings and Engineered Surfaces
Room San Diego - Session E3

Tribology of Coatings for Automotive and Aerospace Applications

Moderators: Sébastien Guimond, Oerlikon Balzers, Oerlikon Surface Solutions AG, Nicolas Argibay, Sandia National Laboratories, Pantcho Stoyanov, Pratt & Whitney, USA

1:50pm **E3-2 Bending Fatigue Property Enhancements of Metallic Substrates by Thin Film Metallic Glass Coatings***, **Chia-Hao Chang**, J Chu,* National Taiwan University of Science and Technology, Taiwan

Thin film metallic glasses (TFMGs) possess extraordinary mechanical properties such as high strength, high toughness, large elastic limit as well as excellent wear and corrosion resistances. Thus, they have attracted industrial interests for the potential applications with their superior mechanical properties. In this presentation, 200-nm-thick TFMG and TFMG/ceramic multilayer coatings were deposited on the 316L stainless steel, Ti6Al4V alloy and ZK60 Mg alloy specimens using radio frequency magnetron sputtering system for four-point-bending fatigue test at room temperature. Fatigue properties of either TFMG-coated or TFMG/ceramic multilayer-coated samples were improved significantly. The hardness of TFMGs and multilayer, their excellent adhesion to the substrate, and the resulting reduction in surface roughness are believed to account for the enhanced fatigue characteristics.

2:10pm **E3-3 Optimization of the Tribological Contact between Piston Ring and Cylinder Wall with Oxide Coatings***, C Bohnheio, P Ernst, P Luethy,* Oerlikon Metco AG, Switzerland*; J Ramm, H Rudigier, **Florian Seibert**, B Widrig,* Oerlikon Surface Solutions AG, Liechtenstein

Strategies to further improve the efficiency of modern combustion engines resulted in the development of so called downsized engines operating at higher power densities and operation temperatures. This implies improved stability of material surfaces in the contact between piston ring and cylinder wall. The piston group contributes also significantly to the energy loss in the powertrain of a combustion engine. Therefore, reduced friction losses are another demand in engine development. The selection of appropriate coatings for the piston ring and the liner surface may respond to both challenges.

Two coating technologies were investigated: physical vapour deposition (PVD) for the piston rings and atmospheric plasma spray (APS) for the cylinder bore surfaces. In a first step, these coatings were applied to test substrates and investigated by a reciprocating wear test (SRV®). The tests were performed under dry and lubricated conditions and for room temperature and temperatures up to 160°C. Wear of the coating and the ball (counter-part) was measured by confocal microscopy. It could be shown that the tribological contact between hard PVD and APS oxide coatings and the alumina counter-part did not only show excellent wear behavior for different surface finishing, but had also the highest stability for elevated temperatures. In a next step, the most promising coating combinations were tested in a motorbike engine configuration and compared with standard materials like CrN for the piston ring and a number of standard coatings for the liner. The tests demonstrated for one oxide-oxide combination an increase of the power output of the engine by 1.5%.

2:30pm **E3-4 Mechanical Characterization of the Glaze Layer formed by Fretting Wear in a Ceramic versus Metallic Alloy Contact***, A Viat, **Gaylord Guillonneau**, S Fouvry,* Ecole Centrale de Lyon, France*; G Kermouche,* Ecole des Mines de Saint-Etienne, France*; J Michler,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland

In a simplified aeronautic blade disk contact, between ceramic and Haynes 25 (cobalt-based alloy) flats, high temperature fretting wear induces third body formation. This third body, called "glaze layer", is created from compacted and sintered debris. Nanocrystalline structure and composition have been described in a previous paper [1]. The glaze layer adhering on both alloy and ceramic counterparts contains counterface material, as observed for ductile contacts [2]. However, the glaze layer does not behave like a ductile material at first sight: under ambient temperature, it is brittle and harder than the two respective substrates.

In-situ SEM micropillar compression at temperatures between ambient and 500 °C have been performed in the glaze layer and its substrates in cross section. This approach focuses on the mechanical behavior at temperatures where the glaze layer is created and is operational for wear protection. The device used is an Alemnis/EMPA high temperature SEM micro-tester [3]. Micropillar compression enables to obtain directly the mechanical response of material surfaces in terms of elasticity, yield stress / hardness and work hardening. Also, the deformation behaviour can be directly observed by SEM images. Firstly, the tribological contact and the device used for in-situ microcompression will be presented. Then the mechanical properties will be detailed and discussed in terms of elasticity and plasticity as a function of temperature. Finally, a discussion about the relation of the glaze layer mechanics with its formation and lubricious properties is proposed.

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2:50pm **E3-5 Sequence of Stages in the Microstructure Evolution in Copper under Reciprocating Tribological Loading***, **Christian Greiner**,* Karlsruhe Institute of Technology (KIT), Germany **INVITED**

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. To cover different stages of this microstructure evolution, high-purity copper was investigated after increasing numbers of sliding cycles of a sapphire sphere in reciprocating motion. Scanning electron and focused ion beam (FIB) microscopy were applied to monitor the microstructure changes. A thin tribologically deformed layer which grew from tens of nanometers to several micrometers with increasing number of cycles was observed in cross-sections. By analyzing dislocation structures and local orientation changes in the cross-sectional areas, dislocation activity, the occurrence of a distinct dislocation trace line and the emergence of new subgrain boundaries could be observed at different depths. These results strongly suggest that dislocation self-organization is a key elementary mechanism for the microstructure evolution under a tribological load. The distinct elementary processes at different stages of sliding identified here will be essential for the future modelling of the microstructure evolution in tribological contacts.

3:30pm **E3-7 Effect of Test Atmosphere on the Tribological Behaviour of the Fluorinated Tetrahedral Amorphous Carbon (ta-C-F) Coatings against Steel***, **MuhammadZafarUllah Khan**, S Bhowmick, A Alpas,* University of Windsor, Canada

Carbon based coatings exhibit low coefficient of friction (COF) in sliding contact against steel, but the resulting COF is highly sensitive to environmental conditions. This study examines the tribological properties of a fluorinated tetrahedral amorphous carbon (ta-C-F)coating containing 12 at. % F (ta-C-F) tested against a 52100 grade steel at 2.0 N and 5 cm/s in ambient air (53% RH), in dry oxygen and in dry N_2 atmospheres with relative humidity reduced to less than 4%. The ta-C-F coating exhibited low and stable steady state COF (μ_s) under ambient (0.25), dry oxygen (0.27) and N_2 (0.22) atmospheres conditions. It was suggested that a carbonaceous transfer layer passivated by F atoms was responsible for low and stable μ_s under all testing conditions as revealed by X-ray photoelectron and micro-Raman spectroscopy. However, the running-in COF (μ_R) varied with the testing atmosphere. The highest running-in COF (μ_R = 0.56) was observed under ambient condition which was reduced to 0.48 in dry oxygen and 0.40 in N_2. The presence of N_2 played an important role in stabilizing carbonaceous transfer layer by limiting the formation of abrasive iron oxide particles.

3:50pm **E3-8 Laser-based Process for Polymeric Tribological Coatings on Lightweight Components***, **Hendrik Saendker**, J Stollenwerk,* Fraunhofer Institute for Laser Technology, Germany*; P Loosen,* Chair for Technology of Optical Systems TOS, Germany

Multiple applications, especially in the automotive sector and in mechanical engineering, are predominantly affected by friction and wear stress and, therefore, represent a substantial challenge for the components being used. Oftentimes, the endurance and the efficiency of these components can be enhanced by means of application-specific tribological coatings. For engine components like pistons or bearing shells made of light metals like Aluminum, current coatings based on sliding lacquer do not meet increasing requirements, particularly regarding temperature resistance and wear protection. Coatings based on high-temperature resisting thermoplastic polymers like PEEK (polyether ether ketone) represents an alternative to conventional tribological coatings. An outstanding challenge results from using temperature-sensitive Aluminum alloy which show structural changes above temperatures of 140 - 180°C, hence below the melting temperature of PEEK of approximately 340°C. In Comparison to conventional oven processes, laser-based processes provide a reduced thermal load of the workpiece. Therefore, the investigated coating process comprises four consecutive steps: a laser-based pre-treatment of the components (1), the preparation of a hydrous dispersion based on PEEK powder (2), the deposition of the dispersion by e.g. spray coating (3), and the melting of the PEEK powder by means of laser radiation (4).

The current investigations are primarily focused on the investigation of the influence of different pre-treatments on the adherence of the coating and the influence of different temporally temperature profiles during laser melting on the properties of the coatings, e.g. relative density, crystallinity, surface roughness and structural changes of the base material. The pre-treatments comprise the processing of the component by means of pulsed laser radiation. For the laser melting of the PEEK powder, different lasers operating in the continuous wave mode are used.

By means of this new coating process, dense and adherent tribological coatings can be applied on Aluminum substrates. The adherence is significantly increased by the laser-based pre-treatment of the metallic substrates.

4:10pm **E3-9 Long-term Tests of Tribological Properties of HVOF-sprayed WCCoCr Carbide Coatings of Ultra-fine Powders with a View to Applying Them to Sliding Rings of Mechanical Seals***, **Aleksander Iwaniak**, R Swadzba,* Silesian University of Technology, Poland*; G Wieclaw,* Certech Sp. z o.o., Poland*; L Norymberczyk,* ANGA Uszczelnienia Mechaniczne Sp. z o.o., Poland

This paper describes tests aimed at exploring the tribological properties of thermal-sprayed coatings. The coatings were deposited onto the face areas of cylindrical metallic specimens whose shape approximated that of sliding rings used in mechanical seals. Long-term (over 400 hours of continuous operation) frictional wear tests of the coated samples were performed at a specialized workstation simulating the operation of a mechanical seal. The wear of the specimens was evaluated by 3D scanning. After the tests, the specimen surfaces were examined by means of a 3D profilometer and a scanning electron microscope (SEM, EPMA). The tests show that WCCoCr coatings sprayed using ultra-fine powders (less than 10 µm) ensure the effectiveness of a friction pair and are characerised by a low degree of frictional wear in comparison with coatings sprayed using powders having coarser particles (25 µm). The highest degree of wear was identified for CrCNiCr-type coatings. The tests indicate that metallic sliding rings covered with WCCoCr carbide coatings of ultra-fine particle powders can be used in certain types of mechanical seals in which solid tungsten carbide (WC) rings are currently used.

Financial support by The National Centre for Research and Development (NCBiR) in Warsaw, Poland - Project No INNOTECH-K2/IN2/2/181798/NCBR/13 is gratefully acknowledge.

4:30pm **E3-10 Role of Oxygen in High Temperature Sliding Behaviour of W Containing Diamond-like Carbon (W-DLC)***, S Bhowmick, M Lou, A Alpas, **MuhammadZafarUllah Khan**,* University of Windsor, Canada

W-containing diamond-like carbon (W-DLC) coating is of interest to the manufacturing industry, as they showed low coefficient of friction (COF) against aluminum at elevated temperatures. The low COF values at 400 ˚C (0.18) and 500 ˚C (0.12) could be attributed to the formation of the transfer layers made of lubricious tungsten oxide WO_3. However, at intermediate temperatures between 100 ˚C and 300 ˚C, a high COF of 0.60 was recorded. In this work, the friction reduction mechanisms of W-DLC coatings were investigated in dry oxygen atmosphere and studied as a function of testing temperature up to 500 ˚C against an Al alloy. The purpose of maintaining an oxygen rich environment was to increase the propensity of WO_3 formation at the sliding surfaces. An average steady state COF (μ_s) of 0.11 was observed at 25 ˚C and low friction values were maintained up to 500 ˚C where μ_s was 0.13. Micro-Raman and X-Ray photoelectron spectroscopy (XPS) revealed that at room temperature the transfer layers were rich in carbon, whereas between 100 ˚C to 500 ˚C the transfer layers primarily consisted of tungsten oxide. The presence of sufficient oxygen in testing atmospheres led to the formation of tungsten oxide rich transfer layers which reduced the COF between 100 ˚C to 500 ˚C. This work shows the importance of compositional characterization and study of mechanisms of transfer layer formation during sliding friction of 319 Al tested against W-DLC coatings.

Tribology and Mechanical Behavior of Coatings and Engineered Surfaces
Room San Diego - Session E1-1

Friction, Wear, Lubrication Effects, and Modeling

Moderators: Albano Cavaleiro, University of Coimbra, Carsten Gachot, Vienna University of Technology, Giovanni Ramirez, Argonne National Laboratory, USA

8:00am **E1-1-1 Stress and Friction Modelling for Improved Nano-scratch Testing of Hard Coatings*, Ben Beake,*** Micro Materials Ltd, UK*; V Vishnyakov,* University of Huddersfield, UK*; T Liskiewicz,* University of Leeds, UK

Nano- and micro-scale scratch tests have significant benefits over the more traditional macro-scale tests with 200 micron end radius diamond indenters. By employing smaller loads and sharper probes it is possible to greatly increase the sensitivity of the test to coating properties. Nevertheless, interpretation of the results can be complex without further analysis. In this paper we present (i) analytical modelling to assess the main stresses acting at the coating-substrate interface (von Mises, tensile and shear stresses) (ii) simulation of load-dependent friction. The modelling has been applied to different coating systems such as DLC on hardened steels and hard nanocomposite TiSiN on silicon. The stress modelling has been able to explain dramatic experimental differences in failure mechanisms with coating mechanical properties due to differences in tensile stresses at the interface and whether plastic flow initiates in coating or substrate. It has been possible to deconvolute the interfacial component of friction from the ploughing contribution and to accurate simulate the total frictional force by additionally considering the load-dependent transfer of load support to the front half of the probe as the deformation becomes less elastic in nature.

8:20am **E1-1-2 Wear Resistance and Solid Lubricity of Nanolayered Molybdenum Containing Nitride Coatings Deposited using Cathodic Arc Technique*, Qi Yang,*** National Research Council of Canada, Canada

Molybdenum containing MoTiN, MoCrN, MoZrN and MoAlTiN nanolyered coatings, were deposited on the 17-4 PH stainless steel substrate by using cathodic arc evaporation technique. Pin-on-disc dry sliding tests were performed to investigate the coatings' wear resistance and solid lubricity. All these coatings, particularly MoTiN and MoAlTiN coatings, demonstrated wear resistance superior to and coefficient of friction significantly lower than the corresponding Mo-free TiN, CrN, ZrN and AlTiN coatings. For example, the specific wear rate of the MoAlTiN coating is less than 0.15% of that of AlTiN while its coefficient of friction is only 0.28 when compared to 0.60 of the AlTiN coating. The excellent tribological performance is contributed to the formation of the surface MoO_3 layer on the wear track due to the tribo-oxidation process. Further wear tests of the MoTiN and MoAlTiN coatings against Al_2O_3 ball revealed less improvement in wear resistance and less reduction in coefficient of friction. When tested against Si_3N_4 ball, both coatings, though showing noticeably better wear resistance than their corresponding Mo-free coatings, did not demonstrate low coefficient of friction. The scanning electron microscopy (SEM) and the energy dispersive X-ray spectroscopy (EDS) analyses of the wear track surfaces illustrated the importance of retaining a stable MoO_3 surface layer in order to maintain the beneficial effect of Mo on the tribological performance of the coatings.

8:40am **E1-1-3 Exploring Tribological Interactions – from Molecules to Engineering Applications*, Daniele Dini,*** Imperial College London, UK **INVITED**

Tribological phenomena are governed by events and mechanisms which find their roots at the small scales, even more so in environments where mechanical and chemical effects are intimately coupled. For example, nano-scale thermal and particle emission events control the formation of antiwear additive films and oxidation; surface damage, such us crack initiation and wear, results from the accumulation of strain at dislocations level; corrosion events are triggered and controlled by molecular interactions. The key challenge addressed in this talk is the need for the development of robust methodologies for the integration of the skills and techniques recently developed by our modelling team at different scales (see e.g. [1-7]) to capture physical, chemical and mechanical processes and interactions across the scales via a multi-physics modelling strategy. Example of modelling methodologies developed and employed to solve problems at specific length- and time-scales will be presented before concentrating on coupling strategies to be adopted to shed light on macro-scale tribological events while zooming-in to understand their governing mechanisms.

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9:20am **E1-1-5 Mechanical Stability under Sliding Contact of Thin Multilayer with Weak Adhesion*, Aymar Quarré de Boiry,*** Joint unit CNRS/Saint-Gobain UMR 125 - Surface of Glass and Interfaces, France*; D Dalmas,* École Centrale de Lyon – Laboratoire de Tribologie et Dynamique des Système, France*; J Faou, J Teisseire,* Saint-Gobain Recherche, France

To modify the surface properties of the flat glass (optical, thermal, electrochemical...), thin film deposition by magnetron sputtering is a technique increasingly widespread. For example, low emissivity glasses which are produced by Glass industry for many years allow reducing energy losses by blocking infra-red. An essential part of those functionalized glasses is a thin silver layer (ten nanometers) deposed by cathodic pulverization. However, these coatings have often low mechanical resistance toward contact loading (scratch) which can happen during manipulation or washing. Scratches are due to contact loading (friction) and are influenced by mechanical properties of the stack (modulus, toughness, adhesion …) [1]. However, a quantitative analysis of the damages is tough because of the complexity and the thinness of the layers (few nanometers). The idea developed during this study is to extend the understanding of the scratch phenomenon in order to improve the scratch resistance of thin multilayers by a method of interfacial patterning. Indeed, it has been recently showed that an alternation of weak adhesion areas (with silver) and strong adhesion areas (without silver) allows improving adhesion [2, 3].

In this study, the objective is to perform ball on plan tests to analyze and understand scratch mechanisms on multilayers thin film with low adhesion deposited on glass. Indeed, the glass transparency allows a direct visualization of scratching process during the tests. First, we focused on homogenous samples with a weak interface and we shows that the scratch is strongly influenced by the presences of brittle layers in the stack. Then, a photolithography protocol is used to pattern a ten nanometers silver layer inside a multilayer stack. In order to understand the scratch phenomenon and the contact between the ball and the layers, many parameters have been measured and their influences and evolution will be discussed: distance between the surface and the sphere, generation of debris, initiation morphology or width of the scratch.

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10:00am **E1-1-7 Tribochemical Investigation of Hydrogenated DLC Films of Different Roughness by Means of Vacuumtribology Accompanied by Mass Spectrometry***, **Matthias Kachel**,* Fraunhofer Institute for Mechanics of Materials IWM, Germany

Shot peening prior to deposition allows to effectively create rough DLC coatings and give them a specific topography. While the effect on the coatings adhesion has been discussed earlier, the main objective of this work is to investigate how a strongly increased roughness influences the tribochemistry of hydrogenated diamond-like carbon coatings (a-C:H).

The shot peening treatment was performed on steel bearing rings using globular shot of WC/Co-88/12 with a grain size of 70 µm. By varying the shot peening parameters, different surface topographies were prepared whose relevant parameters as well as spectral information (PSD) were determined using atomic force microscopy (AFM). The tribological experiments were carried out in a vacuum tribometer (ball on disc) that was equipped with a mass spectrometer. Thus, beside the coefficient of friction, the emission of different hydrocarbons and hydrogen for dry and lubricated conditions were detected. The wear of the ball and the coating was measured after the tribological test via profilometry and was then related to the Z-transition of the ball during the experiment. The structural change of the coating was evaluated using Raman spectroscopy.

The investigantions revealed that the signals obtained by the mass spectrometer correlate directly with the chemical composition of the DLC coating and are therefore a measure of gaseous wear. The ratio of gaseous and solid wear was strongly dependent on the particular surface topography which was found to be responsible for the developement of the COF over time. For the lubricated system, gaseous wear originating from the coating and the oil was detected, giving a measure for oil degradation. In total, the friction coefficient was found to be a function of roughness affecting gaseous and solid wear which both influence the chemical transformation of the coating and the formation of a transfer layer.

10:20am **E1-1-8 Plasma-Assisted Lubrication for the Sliding between Polymer and Diamond-Like Carbon***, S Okumura,* Nagoya University, Japan*; T Hibino, **Hiroyuki Kousaka**,* Gifu University, Japan*; N Umehara,* Nagoya University, Japan

Diamond like carbon (DLC) has widespread applications in many fields due to its excellent mechanical properties such as high hardness, low friction, chemical inertness. Recently, DLC is applied to machine parts as coating to reinforce the surface property. On the other hand, the use of polymer parts made of engineering plastics is increasing instead of metal ones because polymer is light, low cost and easily processed. It is expected in the future that the application of DLC to metal comes to be more frequent in order to further improve the sliding property between metal-polymer contact in sliding parts. For that reason, it is important to clarify the characteristic of friction between DLC and polymer. In this research, silicon doped DLC (a-C:H:Si), which is coated by plasma enhanced chemical vapor deposition (PECVD) on a steel disk (SUS304, JIS) 25 mm in diameter, was used as a mating material of sliding against Polyoxymethylene (POM) which is an engineering plastic. Friction tests were conducted in a roller-on-disk apparatus under dry condition, where the side surface of POM roller 5 mm in diameter and 5 mm in height was contacted to DLC-coated disk at a normal load of 1 N. Sliding test was onducted for 20 minutes at a rotation speed of 200 rpm, where the rotation radius of the roller changes from 5.5 to 10.5 mm along the roller axis. During the total sliding distance of 200 m, helium gas flow and helium plasma flow were irradiated for 50 to 100 m, and 100 to 150 m, respectively. Friction coefficient observed for the first 50 m without any irradiation was around 0.2, which was not changed by the following helium gas irradiation. Then, plasma irradiation caused seriously instable and high friction coefficient (around 1.0); however, after stopping the plasma irradiation, the friction coefficient rapidly fell down to around 0.066 and was stabilized. This result indicates the possibility of plasma-assisted lubrication for the sliding between polymer and DLC. (The authors gratefully acknowledge the funding by JST CREST, Japan.)

10:40am **E1-1-9 Integrated Multiscale Material Modelling of Topographical Effects on Wear and Friction in Sliding DLC Contacts***, K Holmberg, A Laukkanen,* VTT Technical Research Centre, Finland*; **Timo Hakala**,* VTT Technical Research Centre of Finland Ltd, Finland*; H Ronkainen,* VTT Technical Research Centre, Finland*; G Stachowiak, P Podsiadlo, M Wolski,* Curtin University, Australia*; M Gee,* NPL National Physical Laboratory, UK*; C Gachot,* Vienna University of Technology, Austria*; L Li,* Hong Kong City University, Hong Kong

In wear studies, surface topography is typically characterized by standard 2D roughness parameters such as R_a and R_q values. These parameters tend to work well with isotropic surfaces but they are not able to provide full information about surface anisotropy and roughness at different scales of measurement. This limitation of commonly used standard parameters is crucial since most real engineering surfaces are anisotropic and multi-scale objects. Computational modelling and simulation of changes occurring in a material due to surface loading, and calculations of stress and strain in loaded contacts is a research area that is rapidly evolving today. Modelling and simulation of a tribocontact help to understand the mechanisms that result in surface cracking, wear particle formation and wear evolution. 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.

The effect of surface roughness and topographical orientation on surface stresses influencing wear has been investigated for diamond like carbon (DLC) coated steel surfaces with three levels of surface roughness (R_a) in the range of 0.004 – 0.11 µm, and with topographical orientations of grinding marks (grooves) at 0°, 45° and 90°. 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 (VTT Propertune). 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. The macro-topography had a dominating effect on surface cracking, influencing on both wear and friction, while the micro-topographical features contributed to surface cracking by less than 40 %.

11:00am **E1-1-10 A Comparative Study of Fatigue Properties of TiVN and TiNbN Thin Films Deposited On Different Substrates***, **Hikmet Cicek**,* Erzurum Technical University, Turkey*; O Baran,* Erzincan University, Turkey*; A Keles, Y Totik, I Efeoglu,* Ataturk University, Turkey

Transition metal nitrides, especially ternary phase films attract attention due to its high mechanical and tribological features. Besides these, fatigue properties play a very important role on the performance in service life of these type films. TiVN and TiNbN films were deposited on M2 and H13 steel substrates by reactive magnetron sputtering system. Fatigue properties of the films were characterized via multipass scratch tests. 100, 250 and 500 cycle with two directions multipass tests were conducted at room temperature under the 20 N constant load. Structural properties determined with X-ray diffraction, scanning electron microscopy and energy dispersive spectroscopy. Mechanical features of the films were observed with nano hardness tests. Fatigue behaviors, deformation types, coefficient of frictions of the films and effect of different substrates were discussed comparatively. According to the results, TiNbN films showed much better fatigue resistance than TiVN films although critical adhesion load value of TiNbN was less than TiVN film. TiVN films generally showed brittle type cracks at the edge of the tracks though TiNbN films showed more ductile type cracks. Additionally, the films deposited on M2 substrates exhibited better strength than H13 substrates.

11:20am **E1-1-11 The Mechanical and Tribological Properties of Nanocomposite CrMoSixN Coatings***, **Yu-Chu Lu**, J Duh,* National Tsing Hua University, Taiwan

The CrMoN coatings has been found to have superior mechanical and tribological properties, such as high hardness and low friction due to the solid solution strengthening and the formation of lubricating molybdenum oxide. In this study, the mechanical and tribological properties of CrMoN with various Si content was investigated. With Si contents ranged from 0 at. % to 13.0 at. % , the quaternary $CrMoSi_xN$ coatings were deposited on silicon wafer and Inconel 718 by RF magnetron sputter. Through nanoindentation, the hardness and the H^3/E^{*2} ratio of $CrMoSi_xN$ coatings were obtained. The results showed that mechanical characteristics in $CrMoSi_xN$ coatings were strongly influenced by Si contents. The CrMoSixN coating exhibited highest values in hardness and H^3/E^{*2} ratio with 11.0 at.% Si doped, in which columnar grains turned into nanocomposite structure. The strengthening mechanism of nanocomposite structure was attributed to grain refinement and prevention of direct penetration of cracks. Futhermore, the tribological behavior of CrMoSixN coatings were investigated by ball-on-disc tribometer in atmosphere at room temperature, 500 ^{o}C, and 750 ^{o}C respectively. The results indicated that the tribological properties of CrMoSixN coatings could be significantly improved with the Si addition due to protective oxide formation on wear tracks and the cooperation of MoO_3 as solid lubricant. At high temperature, MoO_3 contributed to low friction coefficient, while the low wear rate was owing to the presence of SiO_2. With 11 at.% Si doping, the $CrMoSi_xN$ coatings showed superior mechanical and tribological characteristics, leading to potential applications for wearproof and self-lubricating dry cutting tools at elevated temperature.

11:40am **E1-1-12 Comparative Studies on Tribological Behaviors of a Magnetron Sputtered CrSiN Coating Under the Environments of Air and Water***, **Fangfang Ge**, J Congcong, S Tao, L Peng, H Feng,* Ningbo Institute of Materials Technology and Engineering, Chinese Academy of Sciences, China

Tribological behavior of a magnetron sputtered CrSiN coating with 12.5 at.% Si was studied by ball-on-plate sliding tests in three environments, including air ambient, deionized water, and 3.5 wt.% NaCl water. Then in-situ microscopic examinations on the wear tracks were followed by the combination of scanning electron microscopy (SEM) and a focused ion-beam system. Under the air ambient the coating exhibited a friction coefficient of ~0.47 and a specific wear rate of 1.2×10^{-16} m^3/N m, corresponding to a mild wear process mainly dominated with tribo-oxidation. The tribological behavior of the coating appeared similar features under deionized water and 3.5 wt.% NaCl water. The friction coefficients were relatively lower (~0.24) whereas the specific wear rates increased almost an order of magnitude. It was observed that cracks had formed initially during the running-in period, which might be due to chemical reactions between the coating and the water, resulting more wear of the coating under the environments of deionized water and 3.5 wt.% NaCl water.

12:00pm **E1-1-13 The Mechanical and Tribological Properties of Ti [Nb, V] N Films on the 2024 Al-alloy***, **Ozlem Baran**,* Erzincan University, Turkey*; A Keles,* Ataturk University, Turkey*; H Cicek,* Erzurum Technical University, Turkey*; Y Totik, I Efeoglu,* Ataturk University, Turkey

Al and Al alloys exhibit low wear resistance altough they were used wide range of automobile and aerospace industries. Therefore, in this study, transition metal nitride films were deposited on this light metal alloy in order to improve the wear resistance. The structural properties of the films were analyzed by XRD, SEM and EDS. The hardnesses of the films were determined with a nanohardness test. A pin-on-disc tribometer was used to determine of friction and wear behaviour of the films under different conditions; 50% RH and argon gas. Ti[Nb, V]N films on the Al alloy exhibit a very dense and columnar microstructure. The highest film thickness and the hardness values were obtained as 440 nm and 12 GPa from TiNbVN films. Also, TiNbVN films exhibited the lowest friction coefficent values under different tribo-test conditions. The thickness and the hardness values of TiNbN films are 400 nm and 9.6 GPa, respectively. TiVN films with the lowest thickness (360 nm) and hardness (6 GPa) have the highest friction coefficents under both conditions. The indenter penetration values are 18.75%, 26.4% and 15.23% for TiNbN, TiVN and TiNbVN, respectively. Wear behaviours of the films were affected from the film thickness, hardness and friction coefficent values, significantly.

Tribology and Mechanical Behavior of Coatings and Engineered Surfaces

Room San Diego - Session E1-2

Friction, Wear, Lubrication Effects, and Modeling

Moderators: Albano Cavaleiro, University of Coimbra, Carsten Gachot, Vienna University of Technology, Giovanni Ramirez, Argonne National Laboratory, USA

1:30pm **E1-2-1 Surface Engineering for Increasing Performance of Injection Molding Tools**, ***Lars Pleth Nielsen,*** Danish Technological Institute, Denmark*; S Hengsberger,* Institute of Applied Plastics Research at Engineering College Fribourg, Switzerland*; K Pagh Almtoft, B Hold Christensen,* Danish Technological Institute, Denmark **INVITED**

Injection moulding of high-precision plastic components with high output volumes, using low cycle times without compromising on a high product quality is extremely important in order to increase both the productivity and keeping a competitive edge. At the same time, many moulds are becoming more and more complicated and costly. Hence, it is necessary to increase the lifetime, the wear resistance, minimize the diesel effect, improve the slip properties (ejection force) and the performance of the applied moulds.

The ejection force was quantified in situ during the injection moulding process by incorporating a force sensor. The developed method was found to be so reliable that it was possible to measure a difference between the as machined moulds implying that it was necessary to use each mould as its own reference.

The impact on the ejection force when adding different surface pretreatments to the moulds (as machined, grinded, blasted or laser textured) have been analyzed in combination with different PVD coatings (CrN, HiPIMS CrN, low-temperature pulsed TiAlN) combined with post treatments involving different doses of high-current nitrogen implantation.

The ejection forces were measured for four industrial relevant plastic types (PP, POM, ABS and TPU) before and after adding the wear-resistant coating as well as after ion implantation. The results revealed that the ejection force could be lowered by close to 70% for some of the plastic types by adding a combination of wear-resistant coating and ion implantation. The best performing surfaces were found to be HiPIMS CrN followed by nitrogen ion implantation.

Corrosion and the risk of diesel effect was minimized by Cr ion implantation. The Cr ion implantation was observed to lead to a new phase in the top most 50 nm. The improved corrosion resistance was quantified by cyclic voltammetry.

Thin film characterization of the applied coatings have be addressed based on nanoindentation, SEM and RBS.

The findings will be compared with empirical results from industrial-scale injection moulding.

2:10pm **E1-2-3 Increasing the Lifespan of High Pressure Die Cast Molds Subjected to Severe Wear***, F Silva,* ***Vitor Nunes****, M Andrade,* ISEP - School of Engineering, Polytechnic of Porto, Portugal*; R Alexandre,* TeandM - Technology, Engineering and Materials, S.A., Portugal*; A Baptista,* INEGI - – Instituto de Ciência e Inovação em Eng. Mecânica e Eng. Industrial, Portugal

Despite the increasingly incorporation of composite materials on vehicle components, high pressure die casting still remains one of the most useful manufacturing techniques to obtain automotive parts with complex shape in a cost effective way. It is well known that automotive industry requires high production cadency as well as high products quality. Thus, systematic approaches are permanently being done leading to optimize all the production and management aspects.

The aluminum alloys commonly used in automotive parts such as fuel pumps bodies, throttle bodies, EGR valves, support brackets and so on usually contain Silicon which presents high abrasively. The aluminum flow at high temperature and high speed into the mold induces severe wear, sometimes due to a combination of abrasion and erosion effects.

In this study, two molds with typical severe wear problems were selected and the wear mechanisms involved were deeply studied. After that, a careful selection of the best coating for this purpose was done and some of the most critical parts of the mold were coated in order to test possible effective advantages of the coating application, analyzing the wear resistance behavior and wear mechanisms involved. In parallel, tribological tests were also carried out in order to study if a correlation between laboratorial and industrial tests can be drawn. Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy were intensively used to characterize the coatings and the wear mechanisms observed. Laboratorial tribological tests have involved ball scattering and block-on-ring tests, trying to impose low and medium loads on the contact, respectively. Promising results were obtained allowing to conclude that certain coatings present a better behavior than other ones in this field of application.

Keywords: Wear, Abrasion, Erosion, High-pressure die casting, Mold wear, Wear mechanisms, Mold lifespan

2:30pm **E1-2-4 Effect of Cr Additions on the Structure,Oxidation, Tribological and Machining Performance of Multilayered TiAlN/CrAlN Films Deposited by Sputtering***, F Fernandes,* Instituto Pedro Nunes, Portugal*; M Danek,* Czech Technical University, Czech Republic*; T Polcar,* University of Southampton, UK*;* ***Albano Cavaleiro,*** University of Coimbra, Portugal

Machining of hard to cut materials, such as hardened steels or strong materials for high temperature aerospace applications, is nowadays a challenge of modern engineering. In past recent years, different types of coatings have been developed and applied on the protection of machining tools in order to improve their performance and lifetime. TiAlN has been the most widespread coating because of its sufficient thermal stability, up to 900°C, high hardness, oxidation resistance and adhesion resistance. The addition of Cr to this system has been extensively studied; however, at our knowledge the oxidation, high temperature tribology and in-service machining performance of multilayered TiAlN/CrAlN films is still rare. This work focused on the effect of Cr alloying on the structure, oxidation resistance, kinetics of ions diffusion at high temperature and in-service tribological behaviour of TiAlN/CrAlN films. The results were compared with a TiAlN film deposited as reference. The coatings were deposited in an industrial chamber by unbalanced close field magnetron sputtering, onto Si, FeCrAl alloy and WC substrates as well as onto tungsten carbide drills with 5.5 mm diameter. The crystal structure of the films was analyzed by X-ray diffraction. Oxidation of the films was assessed by thermogravimetric analysis (TGA). Tribological experiments were performed in a high temperature pin-on-disc tribometer at RT, 600 and 700 ºC, using Al2O3 balls as counterpart. The tribological experiments were then complemented with in-real machining tests by studying the lifetime of coated drills. The oxidation performance of coatings is improved with Cr additions due to the growth of a more protective Al-Cr-O rich layer in the interface film/oxide. Tribological behavior of Cr rich coatings at room temperature is similar to the one of reference TiAlN film, but at high temperature it is two to three times better. Coatings with high Cr content (Ti0.28Al0.31Cr0.51N) displayed the best oxidation, tribological and machining performance.

2:50pm **E1-2-5 Investigation on Tribological Behaviour of Boron Doped Diamond Coated Cemented Tungsten Carbide for Cutting Tool Applications***,* ***Ramasubramanian Kannan****, A Narayanaperumal, R Rao,* Indian Institute of Technology Madras, India

In this paper, tribological performance of boron doped microcrystalline diamond (BDD) films and boron doped graded layer diamond thin films (BDD/transition layer/NCD) was studied in detail. The widely used cemented tungsten carbide (WC-Co) was selected as a substrate material for diamond coating. Diamond films was deposited on WC-Co by Hot-filament CVD reactor (HFCVD) setup. Tribology experiment was conducted by using reciprocating tribometer with a normal load of 30 N and a sliding velocity of 10mm/second for a constant wear length of 3 mm. Silicon nitride (Si_3N_4) ball was used as a counter part to study the friction and wear behaviour of diamond films. The surface morphology, topography & roughness of the diamond films were analysed by scanning electron microscope and atomic force microscope respectively. The hardness of the thin diamond films was measured by using berkovich nano indentation test method. The test results found that BDD and boron doped graded layer shows a stable lowest friction coefficient values of 0.004 and 0.003 compared with conventional microcrystalline diamond films (0.007). On the other hand, the wear diameter of the silicon nitride ball for BDD and boron doped graded layer found to be 620 µm and 785 µm, relatively lower in comparison with microcrystalline diamond films (897 µm). The wear track width was measured by scanning electron microscope and shows that BDD and boron doped graded layer indicates lower wear track width 564 µm and 596 µm compared with microcrystalline diamond films (712 µm). Raman mapping was conducted on the wear track of the diamond films to know about the phase pure diamond (sp^3) and partly graphite phases (sp^2) which in turn contributes for the distinct residual stresses in thin films. The obtained lower friction coefficient for boron doped diamond films on WC-Co cutting tool can be suitable for machining of aluminium based metal matrix composites effectively.

3:10pm **E1-2-6 Influence of Self-lubricating Non-metal Phase on the Erosion and Wear Behavior of Ni-based Abradable Coatings***, **Pantcho Stoyanov**, A Wusatowska-Sarnek,* Pratt & Whitney, USA

In this study, the influence of self-lubricating hexagonal boron nitride (hBN) on the erosion and abradability of Ni-based abradable coatings was investigated. Two coatings of same metallic content, one with and one without hBN, were deposited by means of plasma spray to different densities and consequently hardness values. Subsequently, the coatings were subjected to erosion and abradability testing at room temperature in order to evaluate their performance. In addition, to capture the characteristics of the wear process, a detailed chemical and structural analysis was performed within the near-surface region of the worn specimens (i.e. abradable and blade) by means of Scanning Electron Microscopy (SEM), Transmission Electron Microscopy, and micro-Raman spectrometry.

The erosion resistance of the coatings increased with increasing the density as well as with the addition of hBN, which correlated well with the hardness results. The abradability of the coatings without hBN showed a reverse correlation with erosion capability where the less erosion resistant coatings were more abradable as manifested by the lower blade wear. Similarly, the high density hBN content coatings caused higher blade wear compared to the lower density coatings with hBN. However, the coatings with hBN showed overall better abradability compared to the ones without, which correlated well with slightly lower interface temperatures (i.e. measured by means of thermocouples and IR thermometer) and was attributed to the difference in the particle pull-out mechanism.

3:30pm **E1-2-7 Tuning Run-in Friction Behavior of Carbon Film with Graphene Nanocrystallite Structure***, **Cheng Chen**, S Qiu, D Diao,* Shenzhen University, China

In general, amorphous carbon films always has a high friction stage, so-called "run-in" stage, before reaching low friction steady stage. The causes of the run-in stage have been attributed to removal of surface contaminants, oxide film formation, material transfer, and subsurface microstructure reorientation. Regarding to the low friction application, the run-in stage is undesired and necessary to be shortened or even avoided. However, few effective methods have been proposed for eliminating the run-in stage.

In this study, we demonstrated that the run-in stage of carbon film could be tuned with graphene nanocrystallite structure. Firstly, graphene nanocrystallite embedded carbon films were prepared with low-energy electron irradiation using an electron cyclotron resonance (ECR) plasma sputtering system. The structure of graphene nanocrystallite was varied with electron irradiation energy. Friction behaviors of the films were investigated with a Pin-on-Disk tribometer. Compared with amorphous carbon film, the films with graphene nanocrystallite showed shorter run-in stages. And the run-in stage nearly disappeared for the film with irradiation energy of 40 eV. The steady-stage friction coefficients of amorphous carbon film and graphene nanocrystallite embedded carbon films were all about 0.04. The wear rates of the carbon films in run-in stage were measured with a profilometer. The nanostructures of transfer films were investigated by a Raman spectroscopy. The short run-in stage mechanism was interpreted that graphene nanocrystallite was easier to be worn out than amorphous carbon, and it was beneficial for the formation of nanocrystallized transfer film. Secondly, two layer hybrid films were fabricated by depositing 1~10 nm graphene nanocrystallite layer on a thick amorphous carbon layer. The hybrid films showed short run-in stages and low friction behaviors. This study enables a convenient method to control the run-in stage of carbon film, which is significant for tribology application.

3:50pm **E1-2-8 Study of the Wear Mechanisms and Solutions Regarding Inserts used on Cork Grinders***, F Silva, **Thiago Oliveira**,* ISEP - School of Engineering, Polytechnic of Porto, Portugal*; R Alexandre,* TeandM - Technology, Engineering and Materials, S.A., Portugal*; A Baptista,* INEGI - – Instituto de Ciência e Inovação em Eng. Mecânica e Eng. Industrial, Portugal*; A Alves,* Amorim Cork Composites, S.A., Portugal

Cork was one of the main pillars in the Portuguese economy some decades ago, being nowadays one of the most important natural materials currently exported from Portugal to the entire world. Initially, wine bottle stoppers were almost exclusively the only product extracted from the cork oak hull. However, the high quality required by the bottle stoppers makes unviable the use of some cork hooves and the waste generated by the bottle stoppers extraction also is considerable. Moreover, the traditional Portuguese creativity allows bringing to the market a huge number of products based on cork aggregates as composites, due to the addition of bonding and other materials in order to improve the overall characteristics of those products. Nowadays, cork composites are used in products as distinct as sportive floors, wall memos, lady bags or shoes.

However, these composites need to be processed and one of the first steps to produce the cork granules is its grinding process. Despite the cork presenting a relatively low mechanical strength and hardness, the grade of abrasion generated by cork on grinder inserts during the grinding process is considerable. Companies devoted to cork composites have as main initial operation the cork wastes sorting, separating eventual metallic pieces coming to the process together with the cork. Posteriorly those wastes are driven to the grinders leading to the granules generation and further particle size selection. The inserts used in these grinders as main tools to proceed to the grinding process are severely affected by wear and the increasing competitiveness imposed by the market is forcing to face this concern with care.

This study intends to realize what kind of wear mechanisms are strongly influencing the premature end-of-life of the grinding inserts, which occurs due to reduced cutting efficiency and generation of out of specification cork granules, allowing to determine the best ways to extend their life cycle, improving the cost/benefit ratio and allowing to get a better equipment performance by the increase of the OEE (Overall Equipment Efficiency) of the machines related to this manufacturing operation. Results obtained led to understand the phenomena induced in the inserts and some promising alternative solutions using special materials and coatings were drawn and tested, allowing improve the inserts wear behavior thus making this operation more efficient and profitable.

Keywords: Cork, Cork grinders, Grinders inserts, Inserts wear, Wear mechanisms, Abrasion, Coatings

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

EP-2 Clarification of the Relationship between Friction Behavior and Tribo-electrical Performance of Triboelectric Nanogenerator*, W Zhang,* Key Laboratory of Education Ministry for Modern Design and Rotor-Bearing System, Xi'an Jiaotong University, China*; **Pengfei Wang**, D Diao,* Institute of Nanosurface Science and Engineering, Shenzhen University, China

Ever since the concept of triboelectric nanogenerator (TENG) was proposed in 2012, the TENG attracted much attention from researchers for its unprecedented advantages of simple fabrication, low weight, low cost and abundant choices of materials and so on. When the in-plane sliding mode TENG working, one of the friction pair sliding on the other one, sliding friction is introduced to the sliding surface and the friction behavior between two surfaces have great influence on the tribo-electrical performance (including output current, voltage and power) of TENG. However, until now the law between friction behavior and tribo-electrical performance is unclear and it limits the journey of TENG's widely practical utilization. Moreover, the research about the relationship between friction behavior and tribo-electrical performance of TENG isn't receiving enough attention . The target of this research is to clarify the relationship between friction behavior and tribo-electrical performance of TENG.

In this work, graphene sheets embedded carbon (GSEC) films were fabricated by mirror confinement electron cyclotron resonance (MCECR) plasma sputtering method under low-energy electron irradiation and Polydimethylsiloxane (PDMS) are selected as the friction pair materials of in-plane sliding mode TENG. The electrometer and independently designed reciprocating tribometer was used to obtain the output current, voltage and the friction behavior of GSEC film based TENG, respectively. Moreover, the dependence of the short-circuit current and open-circuit voltage on the external loading resistance and the output power as a function of variety resistance of the GSEC film based TENG was obtained with connecting different resistance during measurements. It believed that with the friction coefficient increasing, so did the output current and voltage of GSEC film based TENG, but the energy conversion efficiency decrease. On the contrary, when the friction coefficient decreases, the tribo-electrical performance of GSEC film based TENG decrease slightly but the energy conversion efficiency increase dramatically. The clarification of the relationship between the friction behavior and tribo-electrical performance of TENG is beneficial to improve the tribo-electrical performance as well as optimize the design of TENG.

EP-4 Effect of Cr Content and Various Interlayers on Mechanical Properties of CrAlN Coatings Synthesized by UBMS*, **HoeKun Kim**, J La, M Song, S Lee, Y Hong,* Korea Aerospace University, Republic of Korea

Transition metal nitride coatings are very attractive materials because of their excellent mechanical properties. Especially, the CrAlN coatings have been paid much attention to cutting tool's film due to their high hardness, low surface roughness and excellent thermal stability.In this work the influence of Cr content and various interlayers on mechanical properties of CrAlN coating was investigated. In order to control the Cr content the pulsed DC current was adjusted between 0.4 and 2.0 A, and various interlayers such as CrN, CrZrN, CrN/CrZrSiN were synthesized between the CrAlN coating and the WC substrate. The microstructure, residual stress, hardness and elastic modulus, and friction coefficient were evaluated by field-emission scanning electron microscopy (FE-SEM), laser reflectance system, nano-indentation, and ball-on-disc type wear tester, respectively.

When the Cr content in the CrAlN coatings increased from 0.11 to 0.24 at.%, the hardness and compressive residual stress were measured to be in a range from 31 to 41 GPa, and from 4.3 to 5.7 GPa, respectively. Hardness enhancement could be attributed to the solid solution hardening, in that with Cr insertion. The lattice distortion in the coating developed, and this leads a compressive residual stress enhancement. Therefore, the dislocations became more and more difficult to move, and the hardness of coatings gradually increased. After the scratch test, the critical load of the CrAlN coatings gradually decreased from 48 to 41 N. Generally, the high residual stress causes the low adhesion, and the compressive residual stress could be considered as a factor on the adhesion decreasement. During the wear test, the friction coefficient of the CrAlN coatings with the CrN and CrN/CrZrN interlayer exhibited improved values of 0.34 compared to that of the CrAlN coating with the CrZrN interlayer (COF 0.41). These improved friction coefficient could be attributed to the H/E ratio of the interlayer between the CrAlN coating and the WC substrate. In view of the coating structure, there exists a gradual increase in the H/E ratio from the WC substrate (H/E, 0.040), to the CrN interlayer (H/E, 0.076), and CrZrSiN interlayer (H/E, 0.083), and the CrAlN coating (H/E, 0.089). The CrN and CrZrSiN interlayers induced a smooth transition of the stress effectively under loading conditions, and wear properties could be improved significantly by structuring the coating with an optimal gradient of the H/E ratio of the coating/interlayer/substrate.

EP-5 Effect of Boride Coating on Hydrogen Embrittlement of AISI 8620 Steels*, **MarcoAntonio Doñu Ruiz**, N Lopez Perrusquia,* Universidad Politecnica Del Valle De Mexico, Mexico*; V Cortes Suarez, J Romero Serrano,* Univerisdad Autónoma Metropolitana, Mexico*; M Reyes Cortes,* Universidad Politecnica Del Valle De Mexico, Mexico

The present work studied the effect of boriding coatings on hydrogen embrittlement on AISI 8620 by means the mechanical behavior. The formation of boride were carry out at three different temperatures (1173, 1223, and 1273K) for 6 hours of exposure time by dehydrated paste pack method. After boronizing, the presence of the boride coatings were observed scanning electron microscopy (SEM), X-ray diffractometer and energy dispersive spectroscopy (EDS) analysis.

Hydrogen was introduce into samples with boride coating through cathodic charging applying a current density of 50 mA/cm^2 by 0.5 M sulphuric acid solution kept at a room temperature.

The mechanical behavior of boride coating with hydrogen diffusion were used the following experimental techniques: Vickers micro-hardness, Daimler-Benz Rockwell-C indentation and three-point bend test. As a result of the hydrogen diffusion on sample boride, the borided layer thickness decrease and microhardness tests showed a significant increase in the surface hardness caused by the increased boronizing temperature, moreover the adhesion strength in all condition obtained sufficient cohesion. Finally, three point bend tested show a drastic reduction in ductility and increase the fracture stress value.

EP-6 Characterization and Wear of Co-Cr-Mo-Si Alloy Coatings at High Temperatures*, L Amaral,* Universidade Federal do Paraná, Brazil*; E Nascimento,* Universidade Técnológica Federal do Paraná, Brazil*; **AnaSofia D'Oliveira**,* Universidade Federal do Paraná, Brazil

CoCrMoSi alloys were developed for high temperature applications particularly to resist liquid metal corrosion, due to the distribution of the Laves phase. However, the range of properties of this alloy system allows for uses well beyond the original scope. The metallurgical stability of CoCrMoSi coatings exposed to temperature has been shown to be associated with the stability of Laves phase. The successful use of coatings for high temperature applications requires the understanding of oxidation behavior and influence of the oxide layer on wear. This research focuses on the study of the abrasive wear behavior of CoCrMoSi coatings exposed at 450C and 750C for 6h in an air furnace. The aim is to characterize the oxides formed at the surface of coatings and their role on wear. The CoCrMoSi alloy was deposited by Plasma Transferred arc on AISI304 stainless steel plates (120mmx100mmx12mm). Abrasive wear tests were carried out on a rotating ball apparatus with applied loads ranging from 0.2N to 0,45N. The low loads intended to magnify the impact of surface oxides on wear behavior. For each surface a set of wear tests was carried out with small increments on the applied load until the oxide film was broken and the CoCrMoSi coating started to worn. Raman spectroscopy and X-ray diffraction identified oxides at the surface. Confocal microscopy and scanning electron microscopy analysis characterized the wear scar. At the oxidation temperature used of 450ºC Co and Fe oxides form. The kinects of Co_3O_4, Fe_3O_4 and Fe_2O_3 allow for the fast oxidation of Co and Fe even at low temperatures. Under these conditions scratches were identified on the wear scar associated with the removal of oxide particles, suggesting a low adherence to the coating surface. Exposure at 750ºC resulted on a continuous oxide film of Cr_2O_3 , analysis of the wear scar reveals rolling to be the predominant mechanism. Correlation with non-oxidized CoCrMoSi coatings shows that oxides formed at 450ºC do not impact on the wear performance of coatings. Oxidation at 750ºC resulted on a reduction of friction coefficient, leading to an increase on wear resistance.

EP-7 Influence of Nitrogen Content on the Properties of CNx Coatings Deposited onto AISI H13 Steel by DC Magnetron Sputtering*, Elbert Contreras, F Bolívar, M Gómez,* Universidad de Antioquia, Colombia

The increasing interest for new coatings with higher properties, opened the doors to research of carbon nitrides (CNx); these coatings are attractive for industrial applications due to their its high recovery rates, low friction coefficients and wear rates, in addition to their self-lubricating properties. In this research, CNx coatings were deposited by DC magnetron sputtering, using a Graphite target and a power density of 2,4W/cm^2, deposition temperature was 250°C, working pressure of 6-7x10^{-3} mbar and a BIAS voltage of -70V, prior to the deposition an ionic cleaning was carried out to clean the surface of the substrates, the percentage of nitrogen was varied between 10% and 50% (N_2/(Ar+N_2)) in the gas mixture, in order to evaluate the effect of nitrogen incorporation on microstructure, composition, mechanical and tribological properties. The thicknesses around 2.0 µm were obtained for all coatings; SEM images revealed homogeneous, compact and columnar coatings; the XRD analysis showed that all coatings are completely amorphous. The micro-Raman spectra are characteristic of carbon-rich sample; these clearly show D band (*disordered aromatic rings)* and G band *(graphite).* It was also possible to identify the band associated to C-N triple bonds. The mechanical and tribological properties are affected by the incorporation of nitrogen, by increasing the nitrogen in the gas mixture, increasing the compressive residual stress and hardness; all coatings show similar tribological behavior, with smooth friction records, their low friction coefficients and wear rates are significantly low compared to others self-lubricating coatings like, VN, VSiN, CrVN, etc.

EP-9 Modelling of IN 738 LC Alloy Mechanical Properties based on Microstructural Evolution Simulations for Different Heat Treatment Conditions*, M Boyraz,* ***Bilge Imer,*** Middle East Technical University, Turkey

Conventionally cast nickel-based super alloys, such as commercial alloy IN 738 LC, are widely used in manufacturing of industrial gas turbine blades. With carefully designed microstructure and the existence of alloying elements, the blades show improved mechanical properties at high operating temperatures and corrosive environment. The aim of this work is to model and estimate these mechanical properties of IN 738 LC alloy solely based on simulations for projected heat treatment conditions or service conditions. The microstructure (size, fraction and frequency of gamma prime- γ′ and carbide phases in gamma- γ matrix, and grain size) of IN 738 LC needs to be optimized to improve the high temperature mechanical properties by heat treatment process. This process can be performed at different soaking temperature, time and cooling rates. In this work, micro-structural evolution studies were performed experimentally at various heat treatment process conditions, and these findings were used as input for further simulation studies. The operation time, soaking temperature and cooling rate provided by experimental heat treatment procedures were used as micro-structural simulation input. The results of this simulation were compared with the size, fraction and frequency of γ′ and carbide phases, and grain size provided by SEM (EDS module and mapping), EPMA (WDS module) and optical microscope for before and after heat treatment. After iterative comparison of experimental findings and simulations, an offset was determined to fit the real time and theoretical findings. Thereby, it was possible to estimate the final micro-structure without any necessity to carry out the heat treatment experiment. The output of this microstructure simulation based on heat treatment was used as input to estimate yield stress and creep properties. Yield stress was calculated mainly as a function of precipitation, solid solution and grain boundary strengthening contributors in microstructure. Creep rate was calculated as a function of stress, temperature and microstructural factors such as dislocation density, precipitate size, inter-particle spacing of precipitates. The estimated yield stress values were compared with the corresponding experimental hardness and tensile test values. The ability to determine best heat treatment conditions that achieve the desired microstructural and mechanical properties was developed for IN 738 LC based completely on simulations.

EP-10 Influence of EP Additive Containing Lubricants on the in-situ Formation of Low Friction Tribofilms on Tungsten Based Coatings*,* ***Bernhard Kohlhauser,*** *H Riedl,* Institute of Materials Science and Technology, TU Wien, Austria*; M Ripoll,* AC2T Research GmbH, Austria*; P Mayrhofer,* Institute of Materials Science and Technology, TU Wien, Austria

The reduction of the coefficient of friction combined with reduced wear rates is a major topic in many different industrial applications. Extreme-pressure (EP) and anti-wear (AW) additives as well as low friction coatings have been intensively investigated for several years to achieve this behavior. Lately, the addition of WS_2 or MoS_2 nanoparticles has been proven to be beneficial to the reduction of friction in various tribological contacts. Investigations into the application of sulphur containing lubricants in combination with tungsten functionalized or doped surfaces like W-DLC coatings revealed an additional decrease in the coefficient of friction and wear rates compared to base oils. This effect was proposed to be related to the in-situ formation of a WS_2 carrying low friction tribofilm.

To obtain more information about the suggested formation of WS_2, tungsten based carbide coatings have been deposited applying physical vapour deposition (PVD) and were tested in a linear oscillation SRV testing system. The influence of the applied normal load and EP additive concentration was investigated. Energy dispersive X-ray spectroscopy (EDXS), X-ray photoelectron spectroscopy (XPS), X-ray diffraction (XRD) and transmission electron microscopy (TEM) analyses were carried out to investigate the interface near regions within the tribocontact. An emphasis was placed on the distinction whether the reduction of friction and wear is contributed by the in-situ formation of WS_2 rather than by the generation of Magnéli phase oxides.

EP-13 Laser Cladding Ni-based Alloy/nano-Ni Encapsulated h-BN Self-lubricating Composite Coatings*,* ***Hua Yan****, P Zhang, Q Gao, Y Qin,* Shanghai University of Engineering Science, China*; R Li,* Central South University, China

Nano-Ni encapsulated h-BN/Ni-based alloy (Ni60A) self-lubricating composite coatings on a medium carbon steel were fabricated by laser cladding using two types of lasers: a 5kW continuous wave (CW) CO_2 laser and a 400W pulsed Nd:YAG laser, respectively. A high-energy ball milling method was adopted to clad nano-Ni onto nano-h-BN with an aim to enhance the compatibility between the h-BN and the metal matrix during laser cladding process. The microstructure, phase structure and wear properties of the the self-lubricating composite coatings were investigated by means of scanning electron microscopy (SEM) and X-ray diffraction (XRD), as well as dry sliding wear testing. The research indicated that laser cladding of the self-lubricating composite coatings demonstrates sound cladding layers free of cracks and porosities. It was found that a reaction between h-BN and Ni-based alloy was occurred, which generated hard phase CrB and Ni_3B leading to the increasing of the microhardness of the coatings by CO_2 laser cladding, and laser molten pool suppressed h-BN floating up to upper regions of coating for lower temperature and quickly solidification by YAG laser cladding.The high energy ball milling of nano-Ni onto nano-h-BN significantly improved the interfacial compatibility between h-BN and Ni60A matrix. The friction coefficient of the laser-clad Ni60A/nano-Ni encapsulated h-BN coating was reduced obviously.

EP-14 Leather Treated with Ag/TiO_2 Nanoparticles for Footwear Industry: Tribological and Antimicrobial Activity*, I Carvalho,* University of Coimbra, Portugal*; S Ferdov,* ***CristianaFilipa Almeida Alves,*** University of Minho, Portugal*; M Cerqueira,* INL-International Iberian Nanotechnology Laboratory, Portugal*; R Franz,* Montanuniversität Leoben, Austria*; C Gaidau,* INCDTP-Leather and Footwear Research Institute Division, Romania*; S Carvalho,* University of Minho, Portugal

The proposed work aims to functionalize leathers for footwear industry with antimicrobial properties based on Ag-TiO_2 nanoparticles. The synthesis of nanoparticles was carried out through an innovative and optimized method. The structural characteristics were evaluated by X-ray powder diffraction and the results showed that the TiO_2 nanoparticles are in the anatase phase, with dimensions below 10 nm.

Leather samples were functionalized with TiO_2 and Ag-TiO_2 NP's; these nanomaterials did not change the surface chemical composition of the leathers. Fourier transform infrared spectroscopy showed that the characteristic chemical bands of leathers (-CH_3 stretching vibration) were maintained.

The antimicrobial activity was evaluated by agar diffusion tests against two bacteria species – a Gram negative and a Gram positive, Pseudomonas aeruginosa and Staphylococcus aureus, respectively, and a fungus specie – Candida albicans. The results revealed that the leathers covered with Ag-TiO_2 nanoparticles have antimicrobial activity. The cytotoxicity of nanoparticles was also assessed by MTS test using fibroblast 3T3 which shows the cell's viability. This test was performed to test if these nanoparticles easily penetrated inside the human body. The results showed that the nanoparticles are non-cytotoxic. In order to test the adhesion of the deposited Ag/TiO_2 nanoparticles to the leather substrates, a series of tribological tests in ball-on-disc configuration has been performed using different counterpart materials ranging from rubber (e.g. nitrile rubber) to polymers (e.g. PTFE, PUR or POM). The analysis of the coating wear by light optical and scanning electron microscopy, as well as Raman spectroscopy, revealed details regarding the adhesion of the Ag/TiO_2 nanoparticles depending on the deposition method and parameters applied. The conducted experiments represent a first step towards a systematic study of the mechanical performance of leathers treated with nanoparticles in order to evaluate their suitability for future applications in the footwear industry.

EP-17 An Oliver&Pharr Method for Lateral-Force Nanoindenters*, Norbert Schwarzer,* SIO, Germany

It will be shown how the classical Oliver and Pharr method [1] has to be extended in order to make it fit for the performance and analysis of mixed loading nanoindentation tests. While the classical Oliver and Pharr method can only deal with pure normal loads and allows the extraction of hardness and Young's modulus for a given Poisson's ratio [1, 2, 3], the extended method principally allows for the simultaneous parameter identification of hardness, yields strength in two directions, Young's modulus and Poisson's ration. Under proper experimental conditions, also the extraction of intrinsic stresses seems to be possible. The author will present the method, the theoretical background and a few experimental examples (data from T. Chudoba, ASMEC GmbH, with thanks).

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[2] N. Schwarzer, G. M. Pharr, Thin Solid Films, Vol. 469-470C pp. 194-200

[3] N. Schwarzer, T. Chudoba, F. Richter: „Investigation of ultra thin coatings using Nanoindentation", Surface and Coatings Technology, Vol 200/18-19 pp 5566-5580

EP-18 Investigation of Tribological Properties of Aluminium-Titanium Diboride (Al/TiB_2) MMC under Dry Sliding Condition*, A Sheelwant, S Narala,* BITS Pilani Hyderabad Campus, India*; **Palaparty Shailesh,*** Methodist College of Engineering and Technology, India

Aluminium Metal Matrix Composites are a special class of metal matrix composites with immense potential which open up countless possibilities to enhance properties of materials that are needed in aerospace, military and automotive applications. The potential of these materials lies into their ability to be tailored to fulfil the expectations of the designer. In this study, microstructure and wear properties of aluminium metal matrix composite (AMMC) reinforced with titanium diboride (TiB_2)were investigated. The composite was fabricated through liquid state processing by incorporating 3, 6, 9, 12 and 15 wt% of titanium diboride into aluminium matrix. Uniform distribution of the reinforcement particles into the metal matrix was observed by the microscopic examination of the composite. To determine the friction and wear properties of the composite, experiments were conducted on a pin-on-disc tribometer, under dry condition, by varying applied load and sliding velocity while keeping the sliding distance constant. Loads of 10N, 30N, 50N and velocities of 100 m/min, 200 m/min, 300 m/min were employed over a constant sliding distance. Results obtained from the test revealed that the friction coefficient and overall wear rate increase with the increasing load and sliding velocity. However, the Al/TiB_2 composite shows lower wear rate in contrast to the unreinforced aluminium. Analysis of the worn out surface of the composite under scanning electronic microscope reveals the domination of abrasive wear. The details presented in the current paper form a basis for materials engineers to switch over to AMMCs from monolithic metals and alloys which offer superior wear properties.

Keywords: Aluminium metal matrix composite, Al-TiB_2, Friction, Wear, Dry sliding

EP-20 Tribological Characterization of Thin Films based on Residual Stress, Volume of Wear, Micro-abrasive Wear Modes and Coefficient of Friction*, Ronaldo Cozza, J Wilcken, S Delijaicov, G Donato,* University Center of FEI – Educational Foundation of Ignatius "Padre Sabóia de Medeiros", Brazil

The purpose of this work is to conduct a tribological characterization of thin films based on residual stresses, micro-abrasive wear modes, volume of wear (V) and coefficient of friction (μ). Initially, the residual stresses of thin films of TiN, CrN, TiAlN, ZrN, TiZrN, TiHfC and TiHfCN were analysed by X-ray diffraction; after ball-cratering wear experiments were performed using a ball of AISI 52100 steel and abrasive slurries prepared with black silicon carbide (SiC) particles and glycerine. The normal force (N) and the tangential force (T) were monitored throughout the tests and the coefficient of friction was calculated as $\mu = T/N$. The results showed that the abrasive slurry concentration affected the volume of wear, the occurrence of micro-abrasive wear modes (grooving abrasion or rolling abrasion) and, consequently, the magnitude of the coefficient of friction: *i*) a low abrasive slurry concentration was related with low volume of wear, action of grooving abrasion and a relatively high coefficient of friction; *ii*) a high abrasive slurry concentration was related with high volume of wear, action of rolling abrasion and a relatively low coefficient of friction. In general, the compressive residual stresses measured were relatively low (< 1 GPa).

Keywords: Micro-scale abrasion, residual stress, two-body abrasion, three-body abrasion, PVD coatings.

EP-22 Frictional Behavior of Bismuth-based Soft Coatings*, B Pilotti, G Prieto,* Universidad Nacional del Sur, Argentina*; **Esteban Broitman,*** Esteban Broitman Engineering Consulting, Sweden*; W Tuckart,* Universidad Naciona del Sur, Argentina

The aim of this study was to analyze the frictional behavior of a bismuth-based soft coating, developed using a novel, eco-friendly and economically competitive synthesis. Bismuth has a non-toxic nature, making it attractive for the development of new tribological applications such as coatings or as oil additives.

Bismuth sulfide (Bi_2S_3) nanoparticles were synthesized in-house by means of an eco-friendly reaction in an aqueous medium under mild reaction conditions, in presence of a surfactant. The obtained particles were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The Bi_2S_3 nanoparticles were mixed with a commercial organic varnish in order to generate a soft coating. The coating had a weight fraction of nanoparticles of 17.4 wt% and was manually applied on a SAE 4140 steel disk . The frictional response of the soft coating was evaluated using a pin on disc test (v_l=0.02 m/s; p_0=1100 MPa; 6 m of sliding speed), using an AISI 52100 steel ball with a diameter of 6 mm as the counterpart. The same test procedure was employed using a commercially available molybdenum disulfide varnish to serve as a reference.

Both coatings exhibited similar friction coefficients during the test, with an initial low value (<0.1) that increased slightly towards the end of the test. The bismuth based soft coating showed an average friction coefficient ~30% higher than the molybdenum disulfide coating used as reference.

Note: B. Pilotti, G. Prieto, and W. R. Tuckart are also affiliated to CONICET, Argentina.

EP-24 Compositional and Mechanical Characterization of Ti-Ta Coatings Prepared by Confocal Dual Magnetron Co-Sputtering*, A Bahrami,* Universidad Nacional Autónoma de México, Universidad Nacional Autónoma de México, Mexico*; J Pérez Alvarez, R Mirabal-Rojas,* Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México, Mexico*; **Osmary Depablos-Rivera,*** Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México, Ciudad Universitaria, Mexico*; A Ruiz-Ramirez, A Valencia-Velazco,* Universidad Nacional Autónoma de México, Ciudad Universitaria, Mexico*; S Rodil,* Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México, Mexico

Toughness and hardness are two important characteristics for coating applications in manufacturing industry. In this study, Titanium-Tantalum coating were deposited by magnetron co-sputtering technique, using titanium and tantalum as targets, on steel and silicon substrate. The power applied to the Ti target was fixed at 200 W, while the Ta power was varied from 10 to 60 W. The effects of the Ti- to- Ta ratio on the chemical composition, mechanical and wear properties of Ti-Ta films were investigated. X-ray photoelectron spectroscopy (XPS), Scanning electron microscopy and X-ray diffraction (XRD) were used to evaluate the composition and oxidation state of the films, morphology and structure of obtained thin films, respectively. The hardness was evaluated by nano-indentation test. The XPS results showed that the Ti in the films without Ta is presented only in the metallic state. Also it was found that the percentage of the added Ta varies from 2 to 14 at% by increasing the applied power to the Ta target. The XRD results showed that the coatings were crystalline, and there is no evidence of the formation of intermetallic phases. The results show that increasing the Ta content cause a significant improvement in scratch resistance of the coatings. The crack propagation analysis was evaluated using the data obtained from micro-indentation and the residual stress from XRD and hardness results.

Tribology and Mechanical Behavior of Coatings and Engineered Surfaces
Room San Diego - Session E1-3

Friction, Wear, Lubrication Effects, and Modeling

Moderators: Albano Cavaleiro, University of Coimbra, Carsten Gachot, Vienna University of Technology, Giovanni Ramirez, Argonne National Laboratory, USA

8:00am **E1-3-1 Comparing of Adhesion Properties of TiNbVN Coatings Deposited on Different Substrates**, ***Ihsan Efeoglu**, Y Totik,* Atatürk University, Turkey*; O Baran,* Erzincan University, Turkey*; H Cicek,* Erzurum Technical University, Turkey*; A Keles,* Ataturk University, Turkey

Adhesion properties of hard thin coatings were strong l y affected from used substra t e material. In this study, TiNbVN coa t ings were d e posited on 2024 A l -alloy, M2 and H13 steels substrates using react iv e magnetron sputtering method. Structural and m e chanical properties of the coat i ngs were anal y zed by SEM, EDS, XRD, and nanohardness. Adhesion properties of the coatings were deter m ined via scratch test under progressive load. It was observed that critical load (Lc) values of TiNbVN coa t ings varied in each substra t e. The coati n g m icrostruc tu re is dense a nd the fi l m thickness is about 440n m . The nanohardness values were taken under 1mN load and affec t ed by the substr a tes hardness due to l o ad c arr y ing capacit y . While the softest Al- alloy substrate hardness 1.25GPa, the coating hardness was measu re d 12GPa. On the other hand, the highest coa t ing hardness (39 G Pa) value taken fr o m the hardest M 2 substrate h a rdness (5.7GPa). The results of all the adh e sion tests s h ow a linear relationship between the hardness of th e base mater i al and the adhesion values. While adhesion value fr o m the co ated M2 w a s Lc≈65N, as function of the substrate eff e ct, the measured adhesion values are Lc≈15N and Lc≈50N from 20 2 4 Al-alloy and H13 st e el respectively.

8:20am **E1-3-2 Buckling of Ductile Thin Films on Rigid Substrate**, ***Nadia Ben Dahmane**, G Parry, R Estevez,* SIMaP, University of Grenoble Alpes, CNRS, France*; C Coupeau,* Institut P', Université de Poitiers-UPR 3346 CNRS-ENSMA, France

The process of deposition of thin films can induce large compressive stresses (up to a few GPa) that combined with the low adhesion of the film to the substrate may result in simultaneous buckling and delamination of the film, leading experimentally to a large variety of buckled structures including straight-sided buckles, telephone cords or circular blisters. The mechanism of nucleation and propagation of elastic blisters has been widely investigated and described, either with analytical [1,2] or numerical methods [3]. In particular, the relationship between the mode mixity dependent interfacial toughness on the morphology of the wavy buckles has been highlighted [3]. The response of ductile thin films deposited on rigid substrates remains an open issue. For instance, it has been evidenced experimentally [4] that circular blisters exhibit folding angles at their base larger to that estimated from an elastic model (figure s1). In addition, recent experimental observations of 400nm gold films deposited on silicon wafers showed straight buckles with higher deflections compared to elastic predictions (figure 2). These differences in morphology are thought to originate from the elastic-plastic response of the film but the governing features need to be clarified. This is the purpose of the present study.

In this context, we carry out Finite Element simulations with a model that accounts for isotropic yielding and the non-linearity of the film. This approach aims at identifying the elastic-plastic constitutive model that is able to capture the experimental observations. A mode mixity dependent cohesive zone model is used to describe the thin film/substrate interface, that enables us to study the effect of plasticity on the stability and growth of straight and circular blisters.

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[4] Coupeau et al., Thin Solid Films 469 (2004) 221.

8:40am **E1-3-3 Study of Multi-cracking of Brittle Thin Films and Brittle/ductile Multilayers on Compliant Substrate**, ***Ilhem Ben Cheikh**,* CNRS, Université de Grenoble-Alpes, France*; G Parry,* Laboratoire de Science et Ingénierie des Matériaux et Procédés (SIMaP), Université de Grenoble-Alpes, France*; D Dalmas,* CNRS, Laboratoire de Tribologie et Dynamique des Système (LTDS), Ecole centrale de Lyon, France*; R Estevez,* Laboratoire de Science et Ingénierie des Matériaux et Procédés (SIMaP), Université de Grenoble Alpes, France

Thin films coatings are used in many high technology applications particularly in microelectronics devices. Using flexible polymers as a substrate on which thin films (metal, oxides or organic) are bonded can give rise to new industrial applications such as OLED, flexible electronics or flexible photovoltaics devices. The mechanical stability and failure behavior of multilayer structures deposited on flexible substrate has been extensively studied both experimentally and theoretically [1-3]. Several relaxation mechanisms in thin films have been identified such as channel cracks, debonding or buckle delamination.

The objective of this study is to understand the multi-cracking of the silver and/or zinc oxide layers of various thicknesses coated on elastoplastic substrates (ETFE). In the process of cracking many parameters should be taken into account such as the elastic modulus mismatch between the film and the substrate, the plasticity of the substrate and the ductile or brittle nature of the film.

In the literature several experimental and analytical studies can be found. In [4], after experimental investigations, the existence of three different fracture stages was confirmed, the third one being a saturation stage of the cracks density at high strain with large opening of the existing cracks. Conventional models in literature such as Xia & Hutchinson model [3] and the "Shear lag" formalism [1,2] do not account for those experimental observations. We show that taking into account the plasticity in the substrate allows for capturing the crack density at the saturation regime.

To further validate this new model and the experimental observations, we present a numerical study which uses a cohesive zone model for the interface and to simulate the cracking of the film. This model also takes into account the plastic behavior of the substrate (Fig1). The different stages of cracking observed experimentally, including the nucleation stage were simulated (Fig2). A relationship between the properties of the film toughness, the saturation stress level in the film and the saturation distance between the cracks under deformation has been evidenced.

9:00am **E1-3-4 Tribological Behaviors of UHMWPE Composites with Different Counter Surface Morphologies**, ***Yanzhen Wang**, Z Yin, H Li, G Gao,* Shanghai Jiaotong University, China

The influence of counter surface morphologies on hybrid glass fiber (GF) and carbon fiber (GF) filled ultrahigh molecular weight polyethylene (UHMWPE) were studied under various contact pressure and sliding speed against GCr15 steel in dry condition. The goals were to investigate the tribological behavior of GF/GF/UHMWPE composite as a kind of water lubricated journal bearing material. The friction and wear behavior of composites were examined using a pin-on-disc tribometer. The morphologies of the worn surface were examined by scanning electron microscopy (SEM) and laser 3D micro-imaging and profile measurement. The results demonstrated that the counter surface morphologies have significant influence on the tribological behavior of GF/GF/UHMWPE composite. Generally, the wear rate and friction coefficient of composites increase as the increment of counter surface roughness. Also, the sliding speed and contact pressure have important effect on the tribological behavior of GF/GF/UHMWPE composite. The friction coefficient increases as the increment of sliding speed, while decrease as the increment of contact pressure.

9:20am **E1-3-5 Evaluation of Friction and Wear Characteristics of Electrostatic Solid Lubricant at Different Sliding Conditions**, ***Rakesh Kumar Gunda**,* BITS Pilani Hyderabad campus, India*; S Narala,* BITS Pilani Hyderabad Campus, India

In modern industry, mechanical parts are subjected to friction and wear, leading to heat generation, which effect the reliability, life and power consumption of machinery. Solid lubricant additives have demonstrated better tribological performance in terms of reducing the machining zone temperature by creating friction without polluting the environment. With an appropriate application of solid lubricant additives in the sliding interface, the friction reduction and wear resistance properties of the lubricant have been successfully improved. Therefore, an attempt has been made in this research work with an investigation of using molybdenum disulphide suspension to reduce the friction at machining zone. To achieve this, in the present work, Electrostatic charged spray lubricant (ECSL) system has been envisaged for effective supply of solid lubricant mixture at an extreme low flow rate to the sliding interface of WC pin and Ti-6Al-4V alloy as disk materials. Excessive tribological measurements with SAE 40 oil concentrated with 20wt% of MoS_2 with micron size particles showed friction coefficient as low as 0.001 and negligible wear. It is proposed that negatively charged sprayed MoS_2 solid lubricant mixture at nozzle tip has found remarkable influence on their tribological behavior.

9:40am **E1-3-6 Evaluation of Friction and Wear Properties of Al-TiC_p Metal Matrix Composite under Cryogenic Condition**, ***Sravan Josyula,*** BITS-Pilani, Hyderabad Campus, India*; S Narala,* BITS Pilani Hyderabad Campus, India

Aluminium reinforced titanium carbide particulate (Al-TiCp) composites has gained attention for several engineering and structural applications due to its unrivaled properties, such as high specific strength to weight ratio, high thermal conductivity and excellent wear resistance. However application of Al-TiCp composites in sliding components creates larger friction and wear due to high abrasive nature of reinforcement particles in the soft matrix. It is well known that friction generates increase in contact temperatures can have an imperative impact on the tribological behaviour and failure of sliding components. Further, controlling of friction and wear behaviour enhances the wide application of Al-TiC metal matrix composites in various industrial applications. In this connection current research work try to improve the performance of Al-TiCp composite using cryogenic coolant. Sliding wear tests are performed in order to ascertain friction and wear properties of Al-TiCp metal matrix composites under cryogenic condition. To supply cryogenic coolant effectively to the sliding interface zone, a customized cryo-tribo setup has been developed by fastening cryogenic setup to pin-on-disc tribometer. Comparative studies have been carried out under cryogenic liquid (Liquid Nitrogen (LN_2)) and dry environment under different applied loading and sliding velocities The present analysis reveals that the weight loss of composite sample increases linearly with increase in normal load and sliding distance whereas decreases with increase in sliding velocity. Auxiliary observations revels that presence of cryogenic liquid in sliding contact offers significant reduction in friction and wear values when compared to dry condition through reduction in contact zone temperature and favorable change in pin and disc interface. Microscopic analysis has been carried out to understand the wear behavior of developed under cryogenic condition. The developed technology helps to improve the tribological properties (reduction in friction and wear) there by improving service life and durability of the component in various industrial applications.

10:00am **E1-3-7 Wear Mechanisms and Tribological Characterisation of Novel Nanocomposite Coated Cutting Tool Material for High Temperature Applications**, ***Pavandatta Jadhav,*** *S Narala,* BITS Pilani Hyderabad campus, India

The parts which undergo dry sliding encounter high friction and wear which affects the durability. In recent years, there is a growing interest in the application of Nano-composite coating on the cutting tools to increase the wear resistance, high thermal stability, hardness and durability of the tool. The Nano-composite coatings have been found to have better practical performance capabilities. To reduce the friction and enhance the wear resistance, the hard Nano composites (ZrO_2 Y_2O_3, Ti, Si) N show high performance capabilities. This article attributes a specific study of the application of Nano-composite coating on the cutting tools which shows an impact on the increase in wear resistance, low friction, and increase durability of the tool. In this study, the (ZrO_2 Y_2O_3, Ti, Si) N hard nanocomposite coatings have been deposited on a carbide tool by electrostatic spray coating (ESC) technique. The coatings have been tested for wear and friction behaviour by using a pin-on-disc tribological tester designed according to ASTM G99 standards. The dry sliding wear test was performed on Titanium alloy (Ti6Al4V) disc and (ZrO_2 Y_2O_3, Ti, Si) N hard nanocomposite coated carbide tool (pin) at various speeds and loads at ambient atmosphere. The results revealed that electrostatic Nano-composite coated tools performed much better as compared with those uncoated tools.

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