Tribology and Mechanical Behavior of Coatings and Engineered Surfaces
Room Town & Country B - Session E3-TuM

Coatings for Automotive and Aerospace Applications
Moderators: Nazlim Bagcivan, Schaeffler Technologies GmbH & Co. KG, Germany, **Rainer Cremer**, KCS Europe GmbH, Germany, **Philipp Grützmacher**, Institute of Engineering Design and Product Development , Austria

8:40am **E3-TuM-3 Thermomechanical Stability of Hard DLC Coatings Produced by HiPIMS-DOMS***, **João Carlos Oliveira (joao.oliveira@dem.uc.pt),*** University of Coimbra, Portugal*; A. Vahidi,* University of Coimbra, Iran (Islamic Republic of)*; F. Ferreira, R. Serra, A. Cavaleiro,* University of Coimbra, Portugal

DLC films are very resistant to abrasive and adhesive wear making them suitable for applications that experience extreme contact pressure. For this reason, they are one of the most promising solutions for application in piston´s rings of internal combustion engine (ICE). However, recent trends in the automotive industry, such as reduced engine sizes and turbocharging, require higher operating temperatures and loads, which the classical DLC films deposited by magnetron sputtering cannot withstand. High Power Impulse Magnetron Sputtering (HiPIMS) has been actively investigated for hard DLC deposition. In HiPIMS, a large fraction of sputtered atoms is ionized, thanks to 2–3 orders of magnitude higher plasma densities than in classical magnetron sputtering. However, in the standard HiPIMS process based on Ar, the ionized fraction of C is very low (few percent), due to the low carbon ionization cross section by electron impact.

In previous work, the authors have shown that adding Ne to the plasma up to 50 % results in the deposition of denser and smoother DLC films, with improved tribological properties and increased hardness. In this work, the thermal stability of DLC films deposited in Ar-Ne discharge gas by Deep Oscillation Magnetron Sputtering (DOMS), a variant of HiPIMS, was investigated. In a first step, the thermal stability of the films by annealing up to 700 ºC in a protective atmosphere. The structure of the DLC films deposited without Ne in the discharge gas was stable up to 500 ºC, while clear signs of graphitization were detected at higher temperatures by Raman spectroscopy. The stability of the film up to 500 ºC was confirm by pin-on-disk tests and nanoindentation at room temperature after annealing. The structure of the DLC films deposited with Ne in the discharge gas was stable up to annealing at 700 ºC as shown by Raman spectroscopy. However, the hardness of the films decreases from 25 to 20 GPa upon annealing at 700ºC, while the specific wear rate increases from 0.5 to 1.25 x 10^{-16} m^3/Nm. The thermal stability of the DLC films was also characterized by pin-on-disk tests at high temperature. The coatings were tested up to 400 ºC in ambient atmosphere. Adding Ne to the deposition plasma resulted in an increased thermal stability by 50 ºC for the film deposited with a mixed Ne + Ar discharge gas.

9:00am **E3-TuM-4 Static and Dynamic Friction Assessment Using Novel High Temperature Tribometer***, **Marwan Azzi (marwan.azzi@polymtl.ca),*** Polytechnique Montreal, Canada*; E. Bitar-Nehme,* Tricomat inc, Canada*; J. Sapieha,* Polytechnique Montreal, Canada*; l. Martinu,* Polytechnique Montréal, Canada

Tribology in extreme environments has recently gained significant interest in the aerospace and energy generation communities, largely due to the increased demand for development of durable and more efficient engineering components with an increased performance beyond the current limits. For instance, the next generation gas turbine engines are required to reduce fuel consumption and pollution emission by significant amount which requires a step change in the design and operating environment of the mechanical systems (e.g. higher temperature and contact pressure).

In the present work, a novel experimental test rig has been developed to rigorously investigate the evolution of the static and dynamic friction as well as the wear resistance of a tribological contact exposed to high temperatures (HT) for long period of time. Pin-on-Flat configuration has been adopted in the design with the flat sample being mounted in a furnace that heats up to 800oC. Here, we present an approach to assess static and dynamic friction, and we illustrate it by the results obtained on several materials including stainless steel and Inconel substrates with TiN- and CrN-based vacuum coatings deposited by magnetron sputtering. The measurements showed that a number of sliding cycles is necessary to reach a stable static and dynamic coefficient of friction (CoF). These sliding cycles could be performed uni-directionally or bi-directionally with no effect on the steady-state value of CoF. In addition, it was found that the HT exposure time with closed and stationary tribological contact increases significantly the static CoF which might be related to processes such as diffusion that take place at the interface. The HT tribological testing combined with detailed microstructural characterization of the wear scar (SEM, EDS and Raman spectroscopy) allowed to determine the friction and wear mechanisms. The oxide layer formed at HT was found to play a crucial role in the evolution of HT static friction.

9:20am **E3-TuM-5 Study of the a-C:H Coating Wear Behaviour in Boundary Lubricated Tribological Contacts Using Raman-Based Profilometry (Virtual Presentation)***, **Ardian Morina (A.Morina@leeds.ac.uk),*** University of Leeds, UK*; N. Xu,* University of Leeds, UK , UK **INVITED**

While for ferrous boundary lubricated tribological systems, it is relatively well established that the tribofilms formed from lubricant additives determine friction and wear performance, the effect of lubricant additives on DLC coating wear performance and mechanisms is still not clear. The ability to quantify coating thickness as a function of testing time has the potential to provide new insights on the correlation between tribofilm formation and coating wear, enabling improved lubricant and coating designs.

In this paper, the development of a novel method for measuring coating thickness at the nanoscale level, with the potential to be used for in-situ wear measurement during the test, will be reported.The method is based on using a Raman-active coating underlayer as a sensor of the coating thickness.In this approach, the a-C:H coating is considered a light attenuating layer of the silicon underlayer. The method has been used to study the a-C:H coating wear rate from dry and a boundary lubricated system using molybdenum dialkyldithiocarbamate (MoDTC) additive. A two-stage wear progression mechanism has been proposed for the first time to clarify the detrimental effect of MoDTC-derived tribofilm on a-C:H wear by combining detailed structure and composition analysis. The results have also been supported by post-test Raman spectroscopy, optical profilometer, EELS and TEM analysis.

Keywords: wear measurement, coating, additives, Raman, solid-liquid lubricating

10:00am **E3-TuM-7 Erosion Resistance of TiAlN Coatings for Aerospace Applications***, **Zeliha Idil Kara (e194125@metu.edu.tr),*** S. Ozerinc,* Middle East Technical University, Turkey

Aircraft engine components are subjected to extreme conditions of stress and temperature, resulting in challenging materials requirements. An additional issue is erosion taking place in the air intake since the stochastic nature of the erosion makes the surface susceptible to the formation of micro-cracks, which can cause premature failure that can be catastrophic for the whole aircraft. An effective route to increase the resistance of the components against these harsh environments is the application of wear-resistant coatings. TiN & TiAlN-based coatings produced by physical vapor deposition are commonly preferred due to their low thickness, the ability to effectively coat complex shapes, and the additional advantage of providing resistance to corrosive environments. This study evaluated the characteristics and the performance of TiAlN PVD thin film coatings with an emphasis on erosion performance. Microstructural and mechanical characterization combined with erosion and corrosion testing provided insight into the performance of these coatings under the above explained harsh conditions. Single-layer (≈10 µm thick) TiAlN coatings were applied on the titanium substrates by the cathodic arc deposition method. The microstructure of the coatings was investigated by SEM and X-ray diffraction. The mechanical properties were determined by nanoindentation testing. The adhesion of the coating was examined by scratch tests performed according to EN ISO 20502 in a 10 to 50 N loading range. Solid particle erosion tests were performed according to ASTM G76 by using two different angles of particle impingement of the TiAlN thin film coating. Lastly, salt spray tests were performed according to ASTM B117 to quantify the corrosion resistance of the coatings. The results show that the TiAlN coatings have a high density and exhibit a cubic crystal structure with nanograins. The hardness and elastic modulus of the coatings were in agreement with the previously reported literature values. The erosion tests showed that the coatings dramatically reduce the erosion rate compared to uncoated specimens. The uncoated specimens showed severe surface roughness and crack formation whereas the coated surfaces were intact. Overall, the results demonstrate the effectiveness of the TiAlN coatings in reducing erosion and corrosion, making them suitable for aircraft engine applications. Future studies will focus on the further optimization of the coating composition and morphology for superior mechanical performance.

Tribology and Mechanical Behavior of Coatings and Engineered Surfaces
Room Town & Country B - Session E1-1-TuA
Friction, Wear, Lubrication Effects, and Modeling I

Moderators: Noora Manninen, Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein, **Andreas Rosenkranz**, Andreas Rosenkranz, Universidad de Chile

1:40pm **E1-1-TuA-1 2D Transition Metal Carbide MXenes: Their Synthesis, Tunable Compositions and Mechanical Properties***, **Babak Anasori (banasori@iupui.edu)**,* Indiana University-Purdue University , USA*; B. Wyatt,* Indiana University-Purdue University, USA **INVITED**

Two-dimensional (2D) transition metal carbides, nitrides, and carbonitrides, known as MXenes, have evolved as competitive materials and fillers for developing composites and hybrids for different applications. MXenes are denoted by a chemical formula of $M_{n+1}X_nT_x$ (n = 1 to 4), where M represents n+1 layers of early transition metals (groups 3 – 6 of the periodic table) which are interleaved by n layers of X, where X represents carbon or nitrogen. In addition, T_x represents surface terminations bonded to the outer M layers of MXenes, where T are generally a mixture of –O, –F, –(OH), or –Cl surface groups. MXenes are synthesized via a top-down topochemical etching of their precursor carbides and nitrides, such as MAX phases. MXene flakes have strong mechanical properties (330 ± 30 GPa and 386 ± 13 GPa for $Ti_3C_2T_x$ and $Nb_4C_3T_x$, respectively), which make MXenes the stiffest solution-processable 2D nanomaterials to date. The current forty synthesized MXene compositions paired within-depth ability to control their composition and structure makes MXenes a unique family of 2D materials with unlimited number of compositions and tunable properties. In this talk, we provide an overview of different MXenes compositions, their synthesis, and MXenes' mechanical properties and discuss the effects of MXenes' compositions, synthesis, and processing steps on their mechanical properties.

Keywords: MXenes, 2D materials, carbides, mechanical properties, composites.

2:20pm **E1-1-TuA-3 Grain Boundary Sliding and Low Friction in BCC Metals***, **Michael Chandross (mechand@sandia.gov)**,* Sandia National Laboratories, USA*; A. Hinkle,* CCDC & CBC, Aberdeen Proving Ground, USA*; M. Jones, P. Lu,* Sandia National Laboratories, USA*; N. Argibay,* Ames Laboratory, USA **INVITED**

We show evidence of low friction in BCC metals through molecular dynamics simulations and ultra-high vacuum experiments. This is shown to be correlated with grain boundary sliding (GBS) as the primary mechanism of deformation. Specifically, when grain sizes at the sliding interface are smaller than a critical, material-dependent value (on the order of 10-30 nm), a crossover occurs from dislocation mediated plasticity and Hall-Petch strengthening to GBS and interfacial softening. Results from simulations and experiments are quantitatively compared to a new predictive model of shear strength.

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3:00pm **E1-1-TuA-5 Evaluation of Tribocoatings in Low Viscosity Fuels***, **Maddox Dockins (maddoxdockins@gmail.com)**, A. Ayyagari, S. Srivilliputhur,* University of North Texas, USA*; S. Berkebile,* US DEVCOM Army Research Laboratory, USA*; D. Berman, A. Voevodin, S. Aouadi,* University of North Texas, USA

M. Dockins[1], A. Ayyagari[1], S. Srilliviputhur[1], S.P. Berkebile[2], D. Berman[1], A.A. Voevodin[1], S.M. Aouadi[1]

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Degradation of sliding surfaces creates a significant problem for mechanical assemblies. In the case of next generation fuel delivery systems, steel on steel contacts in low viscosity fuel environments have been shown to experience scuffing failure after extended operation under rough conditions, as well as poor general tribological performance. To prevent this scuffing-induced failure, various surface modification techniques were implemented, such as the application of carbide- and nitride-based protective coatings via PVD and plasma spray methods. These surface modification techniques were evaluated in various fuel environments and counterbody compositions using a high frequency reciprocating rig tribometer in a sliding velocity range of 0.2 to 0.6 m/s and contact pressures ranging from 500 to 1200 MPa. Their relative performance was evaluated according to their wear rates, average coefficient of friction, and oxidation as determined via SEM/EDS. These results were explained theoretically through density functional theory calculations that quantify the effect of surface interactions with the fluid.

4:00pm **E1-1-TuA-8 Phototribology: Control of Friction by Light***, B. Perotti,* UCS, Brazil*; A. Cammarata,* Czech Technical University in Prague, Czech Republic*; F. Cemin,* Université Paris-Saclay and UNICAMP, Brazil*; S. Sales de Mello,* UCS and UNICAMP, Brazil*; L. Leidens,* UCS, Brazil*; F. Echeverrigaray,* UCS and UNICAMP, Brazil*; T. Minea,* Université Paris-Saclay, France*; F. Alvarez,* UNICAMP, Brazil*; A. Michels,* UCS, Brazil*; T. Polcar,* University of Southampton and Czech Technical University , UK*; **Carlos Figueroa (cafiguer@ucs.br)**,* UCS, Brazil

Friction phenomenon is a complex manifestation of nature originated in energy dissipation events owing to the mechanical lost work of non-conservative forces. It is a property influenced by contact area, normal force, surface chemistry, mechanical properties, among others. There are several ways of tuning friction, all of them nonreversible processes. Thus, the active control of friction though external sources is a challenge in tribology. In this study, we report active control of friction forces at the nanoscale in TiO_2 thin films (anatase) obtained by HiPIMS as a function of the presence or absence of UV radiation (λ = 365 nm and nominal power of 5 mW) by friction force microscopy (FFM). According to the effects, this phenomenon of light-matter interaction is reversible, stable, and can be tuned/controlled by UV light. The radiation incidence modifies the physicochemical interactions at the sliding interface in TiO_2 thin films bringing on a dramatic reduction of frictional force of up to 61%. To understand the energy dissipation process, the characteristic frequencies of the system were analyzed; to this aim, atomic force microscopy signals were measured by wavelet analysis. The results show that the surface activation by UV light reduces the dissipated energy. *Ab initio* simulations were used to corroborate that the electron excitation augments the electronic density on the material surface. According to these results one can conclude that the reduction in friction is a result of the lower atomic orbital overlapping on the surface. These findings contribute to a new conceptual framework in tribology where light may be defined as a fourth body and the integration of tribology with photonics and optoelectronics providing a promising direction for applications in micro- and nano-opto-electromechanical systems.

4:20pm **E1-1-TuA-9 Development and Evaluation of Self-Lubricating Nanocomposite Coatings for Metal Forming Dies***, **Jianliang Lin (jlin@swri.org)**,* Southwest Research Institute, San Antonio Texas, USA

Die failure in metal forming industry results in substantial losses of time and money. Conventional lubricants are widely used for die release as well as for cooling assistance on the die surface. However, lubrication is difficult at high temperatures. Oxidation and scaling occur on the work pieces that lead to poor surface finish and possible warping of the material during cooling. The aim of the research is to develop a self-lubricating nanocomposite coating system for metal forming die components and investigate the self-lubricating behavior and thermal stability of the coatings at elevated temperatures. The designed coating systems consist of a nanocomposite matrix doped with solid lubricant phases, e.g. noble metals and amorphous carbon. The composite structure offers multifunctionality, including high wear resistance, good oxidation resistance, crack resistance, and self-lubricating properties at elevated temperatures. In this study, different nanocomposite coating matrixes, e.g. TiSiCN and CrAlN, were doped with different levels of Ag. The coatings were deposited by high power impulse pulse magnetron sputtering (HiPIMS) assisted by hot filaments. The elemental composition, phase structure, microstructure, adhesion, and mechanical properties of the coatings were studied by different means. The tribological properties and self-lubricating behavior of the coatings were evaluated using a high temperature tribometer at 700 °C. The thermal stability and thermal shock resistance of the coatings were evaluated using thermal cyclic test by cycling the coatings from RT to 700 °C with a testing cycle up to 1200 cycles. The results showed that the density and adhesion of all coating systems decreased as the Ag content increased in the coatings. The lowest COF of 0.1 was achieved in the coatings at 700 °C. Excellent thermal stability and thermal shock resistance, and high temperature lubricity were observed in the coatings with an optimal Ag content.

Tribology and Mechanical Behavior of Coatings and Engineered Surfaces
Room Town & Country B - Session E1-2-WeM
Friction, Wear, Lubrication Effects, and Modeling II

Moderators: Noora Manninen, Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein, **Andreas Rosenkranz**, Andreas Rosenkranz, Universidad de Chile

8:00am **E1-2-WeM-1 MXenes: A Model Material for Solid Lubricants***, **Philipp Grützmacher (philipp.gruetzmacher@tuwien.ac.at)**,* Vienna University of Technology, Austria*; C. Gachot,* TU Wien, Austria*; S. Suarez,* Saarland University, Germany*; A. Rosenkranz,* University of Chile

Typically, liquid lubricants are introduced between rubbing surfaces in machine elements and mechanical systems, thus minimizing friction and wear. Diminishing oil resources, the need for ever lower frictional losses, as well as higher demands on the lubricants in terms of resistance against extreme conditions such as high temperatures or low environmental pressures push liquid lubricants to their limits. Therefore, focus turns to solid lubricants. Two-dimensional materials with graphene-like structure have gained remarkable attention, because they have demonstrated excellent tribological properties, even when applying only one or a few atomic layers of the material to the contact zone. There are even several studies, which reported friction coefficients (COFs) below 0.01 and, therefore, superlubricious performance. However, the mechanisms of friction reduction and the influence of the materials' structural and mechanical properties on the latter are still not well understood. One of the newest members of the class of 2D materials are so-called MXene, which were discovered in 2011. MXenes, which are 2D transition metal carbides, nitrides, and carbonitrides, with layers a few atoms thick and, thus, represent an entire class of 2D materials. Single flakes of MXene can be described by the chemical formula $M_{n+1}X_nT_x$ (n = 1 to 4). MXenes offer an extreme versatility in terms of composition, layer thickness, and surface terminations. This makes them ideal model materials to study the influence of theses parameters on tribological behaviour. Despite a still small number of publications on the tribological prosperities of MXenes the field is rapidly growing. First studies have already shown very promising results in terms of wear resistance and friction reduction, even reaching the superlubric regime.

8:20am **E1-2-WeM-2 Structural and Nanomechanical Properties of Manganese Phosphate Coatings***, **Esteban Broitman (esteban.daniel.broitman@skf.com)**, Y. Kadin, P. Andric,* SKF B.V. - Research and Technology Development (RTD), Netherlands*; V. Ott, M. Stüber,* Karlsruhe Institute of Technology (KIT), Institute for Applied Materials (IAM), Germany

Manganese Phosphate (MnPh) coatings are nowadays used in rolling bearings applications due to their advantages such as wear resistance, corrosion resistance, improved fatigue life, and anti-fretting performance.

In this work, MnPh coatings with a thickness of about 5 µm were deposited by a chemical conversion process. AISI 52100 steel substrates were placed in a phosphoric acid bath, where an acid-metal reaction took place locally depleting the hydronium (H_3O^+) ions, raising the pH, and causing a manganese phosphate dissolved salt to fall out of the solution and be precipitated onto the steel surface. Analysis of the surface microstructure and composition of the coatings by X-ray diffraction (XRD), Optical Microscopy (OM), Scanning Electron Microscopy (SEM), and Electron Dispersion Spectroscopy (EDS) has revealed a polycrystalline coating with prismatic-shaped crystals, and about 20% content of Mn. The nanomechanical properties, studied by nanoindentation, exhibit a surface with hardness H_{IT} ~ 1 GPa and Young's modulus E_{IT} ~ 50 GPa. A nanoindentation statistical method was used to obtain H_{IT} and E_{IT} value frequencies over a large area of the coating. We demonstrate that the nanoindentation frequency peaks can be correlated to the coating crystalline orientation revealed by XRD.

8:40am **E1-2-WeM-3 Nanoscale Materials for Macroscale Applications: Zero-Friction and Zero-Wear Carbon Films (Virtual Presentation)***, **Diana Berman (diana.berman@unt.edu)**,* University of North Texas, USA **INVITED**

Friction and wear-related failures remain the greatest problems in today's moving mechanical components, from microelectromechanical devices to automotive assemblies and to biological systems. The critical need to reduce and eliminate the tribological failures constitutes the necessity for continuous search of novel materials and lubrication solutions. In this presentation, we overview recent advances in establishing the fundamental understanding of materials interactions at sliding interfaces and use this knowledge as a guide to developing nanomaterials solutions that enhance reliability and efficiency of tribological systems. We evaluate tribological performance of carbon nanomaterials and demonstrate realization of superlubricity regime at macroscale in carbon-based systems. To extend the lifetime of the tribological materials, we demonstrate tribochemically-driven self-replenishment of carbon-based materials inside the contact interfaces, thus, in addition to the superlubricity, enabling a zero-wear sliding regime.

Overall, the findings have not only allowed us to solve some long-standing puzzles, but could also open a new avenue for the development of new concepts and design strategies for next generation of tribologically efficient materials systems.

9:20am **E1-2-WeM-5 Self Lubricant TiSiN/TiAgN Coatings: Room and High Temperature Tribological Behavior***, F. Fernandes, A. Al-Rjoub,* University of Coimbra, Portugal*; **Albano Cavaleiro (albano.cavaleiro@dem.uc.pt)**,* Instituto Pedro Nunes, Portugal

Increasing requirements on high speed and dry cutting applications open up new demands on the quality of cutting tool materials. This is particularly important in the aerospace and automotive industries where easy and premature degradation of the cutting tools is observed during the machining of hard-to-cut materials. Several solutions have been tried to improve the machinability of these alloys, being the application of thin solid films by sputtering techniques the most promising. However, so far, such solution does not yet allow meet the need for high speed machining and green manufacturing required for machining those materials. In this study, novel multilayered TiSiN/Ti(Ag)N with different Ag concentrations were developed with potential to protect the surface of machining tools under the absence of lubrication. In order to estimate their wear resistance at high temperature the coatings were tribologically tested in a pin-on-disc equipment at different temperatures against two different counterparts (Al2O3 and TiAl6V4 balls). For Al2O3 balls, the hardness and reduced modulus determine the tribological performance of the coatings for tests conducted at room temperature (RT). At 550 ºC, the TiSiN/TiN coating failed, whereas the Ag-containing coating performed better due to the presence of Ag in the contact, which decreased the shear stress and, consequently, the friction. For tests against TiAl6V4 balls, the Ag-containing coating was always better than the TiSiN/TiN one. At 550 C, Ag in the wear track prevented the adhesion of the oxidized Ti-alloy wear debris in the contact, favoring the adhesion of wear debris from the coating to both the coating and counterpart surfaces. No wear could be measured for the 700 ºC tests for both coatings due to different reasons: (i) the presence of oxidized adhered material from the ball to the reference TiSiN/TiN coating surface protected from wear and (ii) the presence of Ag-agglomerated particles decreased the friction and minimized the adhesion wear of the counterpart for the TiSiN/TiN(Ag) coating.

9:40am **E1-2-WeM-6 Design and Tribological Characterization of Self-Lubricating Alloys for Laser Deposition Processes***, H. Torres,* AC2T Research GmbH, Austria*; **Carsten Gachot (carsten.gachot@tuwien.ac.at)**,* TU Wien, Austria*; M. Rodriguez Ripoll,* AC2T Research GmbH, Austria

This work presents the development of self-lubricating metallic alloys for laser deposition processes. Laser deposition processes such as laser metal deposition or direct energy deposition are additive manufacturing techniques that offer a great flexibility and efficiency compared to traditional subtractive manufacturing processes. However, the extreme thermal conditions during deposition and the rapid cooling times pose great challenges in the alloy design. This work illustrates these challenges using metals, such as iron and nickel-base alloys, incorporating lubricious soft metals and metal sulfides.

The microstructure and phase composition of the deposited self-lubricating alloys are characterized using X-ray diffraction, scanning and transmission electron microscopy, showing the importance of having the soft metal as single phase without forming intermetallic compounds or being in solid solution. Additionally, the role on friction of the metal sulfide composition and stoichiometry formed during the laser deposition process is discussed. Afterwards, their friction and wear performance are evaluated using high temperature tribological tests in air and vacuum.

The results reveal that the self-lubricating laser deposited alloys are able to control friction from room temperature to 600 °C in ambient air and at least until 300 °C in vacuum. In ambient air, the friction reduction mechanism is determined by the soft metal and the metal sulfides. At higher temperatures, the contribution of the soft metal diminishes due to oxidation so that the role of metal sulfides to self-lubrication is dominant. In vacuum conditions, the laser deposited self-lubricating alloys can effectively reduce friction down to 0.25 without the aid of an additional lubricant against martensitic stainless steel at 300 °C. This overall tribological performance makes the presented self-lubricating alloys potential candidates for high temperature forming and space applications.

11:00am **E1-2-WeM-10 On the Tribological Performance of Magnetron Sputtered W-S-C Coatings With Conventional and Graded Composition***,* ***Todor Vuchkov (todor.vuchkov@ipn.pt)****,* Instituto Pedro Nunes, Laboratory for Wear, Testing and Materials, Portugal*; A. Cavaleiro,* University of Coimbra, Portugal

Nanocomposite coatings consisting of an amorphous carbon matrix (a-C) with nanocrystallites of transition metal dichalcogenides (TMDs) embedded in it can provide protection against friction and wear in different operating environments, from vacuum to humid ambient air conditions. Magnetron sputtered W-S-C coatings are part of this group of coatings which shows good adaptive tribological behaviour. One potential issue is the running-in behaviour of these coatings as the formation of lubricious tribofilms is crucial for a good tribological response. These tribofilms are most often rich in WS_2, a lubricious compound that is an exceptional lubricant in inert environments (dry N_2, vacuum). A way to improve the running-in behavior would be to change the chemical composition of the coatings across their thickness. Therefore, we deposited a W-S-C coating with varied carbon content across its thickness. The bottom layers of the film were rich in carbon which improved the hardness and thus the load-bearing capacity. The carbon content was gradually reduced towards the top-most layers and finally, a pure WS_2 layer is deposited. In comparison, coatings with a constant composition of ~30 at. % and 50 at. % of carbon were also deposited. The characterization of the coating included scanning electron microscopy with wavelength dispersive spectroscopy for morphological and a study of the chemical composition. X-ray diffraction for structural analysis. Nanoindentation and scratch testing for hardness and adhesion studies respectively. Finally, tribological studies were performed in ambient conditions, dry N_2 environment and at elevated temperature. The correlation between the tribological results and the physico-chemical properties of the coatings revealed that every coating has an advantageous sliding environment.

11:20am **E1-2-WeM-11 Revising the Role of Oxygen "Impurities" in Tribological and Mechanical Performance of MoS_2 Coatings Under Vacuum and Ambient Air Conditions***,* ***Andrey Bondarev (bondaan2@fel.cvut.cz)****, T. Polcar,* Czech Technical University in Prague, Czech Republic

The $Mo_{32}S_{66}O_2$, $Mo_{33}S_{58}O_9$, $Mo_{24}S_{38}O_{38}$ and $Mo_{24}S_5O_{71}$ coatings were fabricated by unbalanced magnetron sputtering. The increase of oxygen concentration in the Mo-O-S system from 2 to 38 at.% displace sulfur, and changes the structure from columnar crystalline to dense amorphous Mo-O-S. Hardness and Young modulus rise with an increase of O concentration from 2 to 38 at.%. These changes are accompanied by enhancement of the tribological performance both under vacuum and humid air. The triboactivated formation of the crystalline MoS_2 tribolayer from amorphous Mo-O-S occurs inside the wear track as well as on the counterpart surface. In the case of the low amount of S for MoS_2 formation,the tribofilm is preferentially formed on the counterpart surface. In the case of the $Mo_{24}S_5O_{71}$ coating with low S content, the lubricious MoS_2 tribolayer is not formed and the coatings fail the tribotests both under vacuum and humid air. Under vacuum, the lowest CoF of 0.02 is observed for the $Mo_{33}S_{58}O_9$ coating and associated with interfacial sliding between MoS_2 transfer film and wear track. For the $Mo_{32}S_{66}O_2$ and $Mo_{24}S_{38}O_{38}$ coatings, CoF values of 0.05 and 0.04 are recorded, this slightly higher CoF is associated with mixed interfacial sliding/transfer film shearing mode of friction. In humid air the CoF values of the coatings were 0.27, 0.29, and 0.18 for the $Mo_{32}S_{66}O_2$, $Mo_{33}S_{58}O_9$, and $Mo_{24}S_{38}O_{38}$ coatings,respectively. Probably, the friction is controlled by physisorbed water between the MoS_2 tribolayers that forms hydrogen bonds with S atoms which increases the interlayer binding energy, and CoF, respectively. Wear resistance under vacuum is governed by the ability of the coatings to form continuous and stable MoS_2 tribolayer and their mechanical properties. Wear resistance in humid air is governed by cohesive wear that probably takes a place because of increased interlayer binding energy. Important, that in the case of a moderate concentration of O in the coatings composition ($Mo_{33}S_{58}O_9$) the water physisorption can be suppressed, and that improves wear resistance.

11:40am **E1-2-WeM-12 Understanding Ultra-Low Coefficient of Friction of a-C Coated Surfaces Under High Contact Pressure and Humidity Levels***,* ***Newton K. Fukumasu (newton.fukumasu@usp.br)****, A. Tschiptschin, I. Machado, R. Souza,* University of São Paulo, Brazil

Carbon-based coatings have attracted significant attention of aerospace, automotive and energy industries, due to low friction and high wear resistance. Usually, these tribological characteristics can be tailored based on different levels of sp^2 to sp^3 carbon bond ratios induced by the presence of hydrogen/dopants or deposition process variables, such as power, substrate bias and sample to target distance. Literature reports that ultra-low friction and superlubricity behavior of carbon-based coatings are influenced by the presence of hydrogen and water molecules at the contact during the tribological test. In this work, experimental and numerical analyses were carried out to understand the conditions of the a-C contacting surfaces that promote ultra-low friction states under high contact pressures. The experimental analyses consisted of ball-on-disk reciprocating tests to evaluate friction and wear of non-hydrogenated amorphous carbon (a-C) coatings, deposited by pulsed-DC magnetron sputtering technique. A polycrystalline graphite target was used to deposit the a-C coating over AISI H13 disks and AISI 52100 balls. Scanning Electron Microscopy (SEM), Raman spectroscopy, optical profilometry and instrumented indentation were applied to characterize tested surfaces. Tribological tests were carried out under dry contact conditions at room temperature, high relative humidity (>60%) and high Hertzian contact pressures (>2 GPa). Results indicated a correlation of the G band peak position (GBPP) with the coefficient of friction. When both surfaces were coated, results showed a stable ultra-low coefficient of friction, stabilized down to 0.03 for the highest GBPP. The digital tribology platform TriboCODE was applied to observe the stress distribution in coated surfaces under tribological applications, in which multiscale numerical simulations suggested a possible whole of water molecules, between carbon surfaces, that induces localized changes on carbon bonds, forming structured tribo-films, promoting both reduction of friction coefficient and improved wear resistance.

Tribology and Mechanical Behavior of Coatings and Engineered Surfaces

Room Town & Country B - Session E1-3-WeA

Friction, Wear, Lubrication Effects, and Modeling III

Moderators: Noora Manninen, Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein, **Andreas Rosenkranz**, Andreas Rosenkranz, Universidad de Chile

2:00pm **E1-3-WeA-1 Critical Materials-Free Cermet Coatings by Thermal Spraying: Sliding and Abrasive Wear Behaviour (Virtual Presentation)***, Giovanni Bolelli (giovanni.bolelli@unimore.it),* Unimore, Italy **INVITED**

Cermet coatings provide resistance against sliding and abrasive wear in e.g. petrochemical valves, papermaking rolls and blades, pump parts, rotary joints and sliding elements of high-speed automatic machinery, etc.

The better-known compositions are based on WC and/or Cr_3C_2. WC-Co-based compositions possess excellent wear resistance at room and moderate temperatures; however, they present two types of criticalities. W and Co are regarded as critical raw materials by many industrialized countries, due to the risk for supply shortages (primary production being concentrated in few manufacturing countries) together with their strategic relevance. On the other hand, Co is now labelled as probable human carcinogen (cat. 1B). This increases the workplace safety requirements. There might also be risks for end-users if fine particulate debris is released. Synergistic effects between Co and WC increase further the inhalation health risk.

Cr_3C_2-NiCr, employed for high-temperature applications and/or corrosion resistance, could alleviate the issues with W and Co, despite the hazardousness of Ni itself, and the potential release of volatile Cr compounds during spraying. However, its low-temperature wear resistance is no match for WC-based materials.

This contribution summarises the authors' recent research on potential alternatives, following two distinct approaches:

-Coatings from commercially available or experimental powder formulations based on WC with Co-free (or nearly Co-free) matrices based on Ni (e.g. WC-NiMoCrFeCo) and/or Fe (e.g. WC-FeNiCrMoCu, WC-FeCrAl).

-Coatings where both Co and WC are replaced. The work has mostly been focused on TiC-based compositions with matrices such as Ni-20Cr, Fe-Cr-Al, or Fe-Ni-Cr alloys.

Results obtained so far show that, in sliding wear applications and/or under corrosive conditions, WC-based hardmetals with Co-free matrices can be a viable replacement for WC-CoCr, matching or sometimes even surpassing its performance. The sliding wear resistance of TiC-based coatings is intermediate between WC-CoCr and Cr_3C_2-NiCr ones, and they are serviceable up to at least 400 °C. So, they could suit a range of low- to intermediate-temperature applications, when the extreme wear resistance of WC-based materials might not be needed. Drawbacks and limitations to be overcome include the tendency of TiC-based formulations to oxidize during thermal spraying, which induces brittleness and impairs the resistance to coarse particles' abrasion.

2:40pm **E1-3-WeA-3 Tribological Behavior of Zirconium Coated Ti-6Al-4V by Pack Cementation***, **Beyza Öztürk (beyza.oeztuerk@dechema.de)**, L. Mengis,* DECHEMA Research Institute, Germany*; D. Dickes, U. Glatzel,* University of Bayreuth, Germany*; M. Galetz,* DECHEMA Research Institute, Germany

The Ti-6Al-4V alloy is used in aerospace, automotive or biomaterial applications due to its excellent mechanical strength and corrosion resistance as well as biocompatibility. However, its poor tribological behavior limits its applications. In this study, zirconium coatings are produced on Ti-6Al-4V by pack cementation process at 700, 900 and 1050 °C for 5 h with the goal to form ZrO_2 on the surface. After that, coatings are examined by optical microscopy (OM), electron microprobe analysis (EPMA) and X-ray diffraction (XRD) techniques. The wear behavior of uncoated and zirconium coated Ti-6Al-4V against Si_3N_4 is studied under dry sliding conditions in the temperature range of RT-600 °C using a ball-on-disc tribometer. Finally, the wear mechanism of the tribopair is investigated by scanning electron microscopy with an energy dispersive X-ray analyzer (SEM/EDX) and profilometry.

3:00pm **E1-3-WeA-4 Study of Electrochemical and Tribological Properties of Electrophoretic Deposited Thin and Thick Graphene Coatings on Pure Titanium Substrate***, **Madhusmita Mallick (madhusmita1509@gmail.com)**, A. N,* Indian Institute of Technology (IIT), Madras, India

Titanium and its alloys have been used widely in various sectors owing to their exceptional properties of light weight, high strength and biocompatibility. However, their poor abrasion and corrosion resistance behaviors are major cause of concern. To mitigate these problems, graphene protective coatings were applied. Therefore, this study focusses on improvement of both corrosion and wear resistance properties of commercially pure-Ti (grade 2) by electrophoretic deposited (EPD) graphene coatings. The effects of different coating thickness and morphology on the electrochemical and tribological performance were investigated. The test results revealed that the smooth and compact microstructure of thin graphene coating of thickness 2.16 µm exhibited superior corrosion resistance and lubricating properties (COF=~0.02) compared to thick graphene coating of thickness 11.8µm (COF=~0.17). However, thick graphene coating demonstrated higher durability of its lubricating behavior and good wear resistance property. Nevertheless, both graphene coatings showed significant improvement in both corrosion and wear resistance properties of bare Ti substrate for various industrial applications.

Tribology and Mechanical Behavior of Coatings and Engineered Surfaces
Room Town & Country B - Session E2-1-ThM
Mechanical Properties and Adhesion I

Moderators: Carsten Gachot, Vienna University of Technology, Austria, **Bo-Shiuan Li**, Oxford University, UK

10:40am **E2-1-ThM-9 Effect of Thin Film Properties on Delamination Behavior and Interface Adhesion***, Alice Lassnig (alice.lassnig@oeaw.ac.at), S. Zak, R. Pippan,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben, Austria*; C. Mitterer,* Montanuniversität Leoben, Austria*; M. Cordill,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben, Austria

Understanding the factors affecting thin film delamination and quantifying thin film adhesion to their substrates is important to ensure reliable multi-material components which are widely encountered in several technological applications.
Of special interest are brittle-ductile interfaces separating thin ductile films from rigid substrates since they are particularly weak. Standard routes to quantify such interfaces are 4 Point Bending tests or - if applicable- buckle-driven delamination by means of the stressed overlayer technique [1]. Improving the interface stability for nanosized thin films on brittle substrates is therefore crucial to ensure reliable bi- and multi-material devices.

In a recent study [2] we could demonstrate that intrinsic film properties such as grain size can significantly influence interface adhesion. Nanoindentation results allowed to estimate the film yield stress of the films when bonded to the substrate. The mixed-mode adhesion energy for each film ranged from 2.35 J/m^2 for the films with larger

grains to 4.90 J/m^2 for their small-grained counterparts while all the films showed similar residual stresses and same film thicknesses. A newly developed focused ion beam (FIB) cutting technique allowed to decouple and quantify the amount of elastic and plastic deformation stored in the buckled thin film. This surprising result could be understood by introducing a new finite element modelling technique to further help understand plasticity contribution as a toughening mechanism during thin film delamination [3] and to further adapt established models, which currently only account for purely elastic film delamination.

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[2]A. Lassnig, V.L. Terziyska, J. Zálešák, T. Jörg, D.M. Többens, T. Griesser, C. Mitterer, R. Pippan, M.J. Cordill, Microstructural Effects on the Interfacial Adhesion of Nanometer-Thick Cu Films on Glass Substrates: Implications for Microelectronic Devices. ACS Appl. Nano Mater. 4 (1) (2021) 61–70. doi:10.1021/acsanm.0c02182.

[3]S. Zak, A. Lassnig, M. J. Cordill, R. Pippan, Finite element-based analysis of buckling-induced plastic deformation. Journal of the Mechanics and Physics of Solids 157 (2021) 104631. doi: 10.1016/j.jmps.2021.104631.

11:00am **E2-1-ThM-10 Buckling-Induced Delamination: Connection between Mode–Mixity and Dundurs´ Parameters***, Stanislav Zak (stanislav.zak@oeaw.ac.at), M. Cordill,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben, Austria

Modern electronics and micromechanical devices consist of various combinations of layered materials and/or coatings with different material properties. In recent years, differences in the elastic moduli and Poisson's ratios are becoming more pronounced (e.g. when bendable electronic components are designed). Therefore, a strong push to investigate interface stability with a more in–depth view on the elastic material properties mismatch influence is needed. Measurements of the adhesion energy of the thin film on the different substrate materials can be made using the spontaneous (compressive stress induced) buckling method described by Hutchinson and Suo. This method induces lateral deformation of the thin film with respect to the substrate (to form delaminated buckles), which causes a significant shear loading contribution to the delamination crack front and therefore, the inevitable mode II appearance. Additionally, the shear loading can be highly influenced by the differences in deformations of the film and substrate due to their elastic mismatch which can be high for modern material combinations, such as those found in flexible devices. Therefore, the original approach to elastic mismatch by Hutchinson and Suo is revised and extended in this work. Since the elastic mismatch on the interface between two different materials can be described by the Dundurs' parameters, a wide range of parameters are used to evaluate the mode-mixity at the delamination crack front. Finite element (FE) modelling is combined with analytical solutions to evaluate parameters describing the mode–mixity of the delaminated buckles as a function of elastic mismatch (both according to Hutchinson and Suo and also with the use of more real-like models). The FE approach enables relatively simple and quick evaluation of stress intensity factors for arbitrary models via domain integration method. Therefore, several FE model configurations can be analysed together with numerically obtained stress/strain fields in the buckled samples. The resulting map of the mode-mixity as a function of Dundurs' parameters will lead to more precise experimental measurements of the mode I and mode II adhesion energy components for modern material combinations.

11:20am **E2-1-ThM-11 Colored Picosecond Acoustics Versus Scotch Tape Adhesion Test: Confrontation on a Series of Similar Samples With a Variable Adhesion***, A. Vital-Juarez,* IEMN UMR CNRS 8520, France*; J. Desmarres,* CNES, France*; **Arnaud DEVOS (arnaud.devos@iemn.fr)**,* IEMN UMR CNRS 8520, France

The study of the bonding at interfaces between thin layers is of great importance in optical and electronic applications as well as in the field of space exploration. A large number of methods have been developed to characterize the adhesion of a thin film to a substrate. Among them, Scotch Tape test is still one of the most popular methods.

Acoustic waves and especially ultra-high frequency acoustic waves are also sensitive to adhesion defects as they affect the way acoustic waves are reflected at the concerned interface.

In this paper, we prepare a dedicated series of identical thin-film samples with a variable adhesion at the interface between the film and its substrate. For that several avenues have been explored: introduction of a weak layer between the film and the substrate; reinforcement of the weak layer adhesion using ion beam implantation. The samples are then characterized using two much different techniques: colored picosecond acoustics for measuring thickness and acoustic reflection coefficient at the interface and scotch tape test to have an independent evaluation of the adhesion. An excellent correlation is found between techniques regarding adhesion that confirms the capability of APiC to perform adhesion test in a totally non destuctive manner and furthermore locally.

11:40am **E2-1-ThM-12 High-Throughput Screening of Adhesion and Friction of Solid Interfaces***, **Maria Clelia Righi (clelia.righi@unibo.it)**,* University of Bologna, Italy **INVITED**
clelia.righi@unibo.it

High-throughput studies have become a valuable tool for material advancement, which is of tremendous importance for industry and closely tied to various societal challenges like clean energy production. We designed and implemented an advanced workflow, based on the FireWorks platform, to perform first principles calculations using the VASP package. Starting from two elemental solids, the workflow can generate the surfaces of interest and match them to form an interface. The interfacial adhesion, load-displacement curves, shear strength and charge displacements are automatically calculated and the results are stored in a public database [1].

In a first application of the high-throughput approach tohomogeneous interfaces we generated a database of the shear and cleavage strengths for over hundred interfaces formed by matching two equivalent surfaces of different elemental crystals [2]. We discovered the existence of a power-law between the adhesion and the shear strength for these interfaces. Moreover, the ratio of the strengths can provide an estimate of the material failure mode[3]. The analysis also showed that the mechanical properties of elemental crystals are closely related to their electronic structure [4].

In a more recent advancement, we created a database for heterogeneous interfaces focusing in particular to metal-on-metal surfaces relevant for technological applications and 2D layers adsorbed on solid substrates. The analysis of the overall trends and the comparisonof the data obtained for heterogeneous interfaces with the homogeneous counterparts improved the scientific and mechanistic understanding of interface mechanics and tribology [5].

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[2] Restuccia, P. et al., “Ideal adhesive and shear strengths of solid interfaces: A high throughput ab initio approach” Comp. Mat. Sci. 154, 517 (2018).

[3] Wolloch, M. et al., “High-throughput screening of the static friction and ideal cleavage strength of solid interfaces” Scientific reports, 9, 1 (2019).

[4] M. Wolloch, G. Levita, P. Restuccia, M. C. Righi, “Interfacial Charge Density and Its Connection to Adhesion and Frictional Forces,” Phys. Rev. Lett., 121, 2 (2018).

[5] G. Losi, O. Chehaimi and M. C. Righi, ”High throughput screening ofsolid interfaces for tribological applications”, in preparation.

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Tribology and Mechanical Behavior of Coatings and Engineered Surfaces
Room Town & Country B - Session E2-2-ThA
Mechanical Properties and Adhesion II

Moderators: Carsten Gachot, Vienna University of Technology, Austria, **Alice Lassnig**, Austrian Academy of Sciences, Austria

2:00pm **E2-2-ThA-3 Reliability Assessment of Thin Films and Multilayers in Electronic Packages (Virtual Presentation)***, **Golta Khatibi (golta.khatibi@tuwien.ac.at)**,* TU Wien, Austria **INVITED**

The rapid technological advancements and market demands in microelectronic sector requires highly accelerated and practice relevant reliability assessment methods. The devices are composed of a variety of dissimilar materials with different chemical, physical and mechanical properties and length scales. The structural integrity and reliability of the systems is related to the resistance of the constituent materials and their interfaces to alternating thermomechanical loads. Design and fabrication of functional and reliable electronics requires a knowledge of fatigue and delamination behavior of the structures and understanding of the related micro-mechanisms of damage. Recently isothermal accelerated mechanical fatigue testing has been proposed as an alternative to standard thermal cycling procedures for rapid evaluation of microelectronic devices. Dedicated dynamic testing set-ups are designed to simulate the dominating loading conditions in the devices while replacing the thermo-mechanically induced strains with equivalent mechanical strains. As a result, physically meaningful lifetime curves can be obtained and the vulnerable sites of the structures can be detected in a short time.

This talk includes a brief introduction on the application of the proposed method for evaluation of various type of multilayers followed by a case study on the effect processing parameters and environment on cyclic delamination behavior of thin films. The experiments were performed by using a dynamic four-point bending setup working in a broad frequency range, equipped with an environmental chamber. Static and dynamic tests were conducted on suitable sandwich type samples prepared from semiconductor tests structures consisting of Si/SiN/TiW/Cu/Polyimid films.Crack length as well as crack opening displacement was measured intermittently at different loading amplitudes. A fracture mechanics based method was applied to derive the relationship between the delamination growth rate (da/dN) and the cyclic energy release rate (ΔG). It was found that the cyclic interfacial delamination occurred at considerably lower values of energy release rate comparing to the critical adhesion energy of the same interface under quasi static loads. Furthermore, experimentally determined crack propagation rates were explained with a power law depending on the accumulated plastic strain per loading cycle by using FEM simulations.

2:40pm **E2-2-ThA-5 A Measurement Structure for *in-situ* Electrical Monitoring of Fatigue Delamination***, **Sebastian Moser (sebastian.moser@k-ai.at)**, D. Tscharnuter, M. Nelhiebel, M. Reisinger, J. Zechner,* KAI Kompetenzzentrum Automobil- und Industrieelektronik GmbH, Austria*; M. Cordill,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben, Austria

Semiconductor devices are comprised of multilayer material structures that undergo cyclic thermal loading with associated thermal stresses during service. A typical fatigue mode of such devices is delamination, which may result in impairment of the system's capability of passivating from environmental influences. At a certain stage, delamination might result in a loss of functionality in terms of mechanical, electrical, or thermal aspects sometimes leading to catastrophic failure of the device, for example, by means of bursting or local melting. It is apparent that in such a case, an accurate root cause analysis or localization of the failure is very challenging or even not possible. For this reason, the development of experimental methods capable of characterizing early-stage delamination is desired. This work introduces a specifically designed measurement structure that has been integrated into an actively heated microelectronic test chip and allows monitoring delamination *in situ* by means of electrical resistance measurements. The chip consists of a silicon substrate, a thin stack of functional layers, and a comparatively thick copper metallization (20 µm). Application of heat pulses causes significant thermo-mechanically induced strain in the copper due to its mismatch in coefficient of thermal expansion (CTE_{Cu} = 17 ppm/K) with respect to silicon (CTE_{Si} = 2.56 ppm/K). Under well-chosen experimental conditions, this drives cyclical delamination of the copper metallization from a functional barrier layer. The delamination monitoring structure presented is characterized by a layout with distinct geometric features that, when passed by the gradually advancing delamination crack front, result in characteristic responses in the electrically monitored signal. This allows a straightforward interpretation of the measurement data without having to rely on any separate calibration experiments. The functionality of the delamination monitoring structure has been proven, firstly, by a simple model that predicts the experimentally obtained curves very well and, secondly, by focused ion beam investigations at different delamination stages. A representative experimental study is presented quantifying the effect of different heating amplitudes, Δ*T*, and test environments, forming gas versus air, on the delamination rate.

3:00pm **E2-2-ThA-6 Modeling of Residual Stress Evolution in Thin Films: Effects of Growth Kinetics, Microstructural Evolution and Energetic Particle***, E. Chason, T. Su, Z. Rao, S. Berman,* Brown University, USA*; **Diederik Depla (diederik.depla@ugent.be)**,* Ghent University, Belgium

Residual stresses in thin films have significant effects on their performance and reliability.To control it, it is useful to understand how the stress is related to the processing conditions used for the film deposition.We describe an analytical model that we have developed that relates the stress to the physical processes occurring film deposition.The model explains the dependence on the growth conditions, microstructural evolution and energy of incoming particles.We have implemented this model in a least-squares fitting program to determine the kinetic parameters controlling stress.Modeling results are shown for numerous published results in the literature for different materials (Ag, Cu, Ni, Fe, Cr, Ti, Co, Mo) using evaporation and sputter deposition. The model is being developed into a web-based application that others can use to analyze their stress measurements and predict the stress under different processing conditions.

3:20pm **E2-2-ThA-7 Mechanical, Structural, Morphological and Biological Evaluation of Multilayer Coatings of HA-Ag/TiO2/TiN/Ti on Ti6Al4V Obtained by Magnetron Sputtering for Implant Application***, **Julián Andrés Lenis Rodas (julian.lenis@udea.edu.co)**, F. Bolívar Osorio, E. Contreras Romero,* University of Antioquia, Colombia*; A. Hurtado Macías,* CIMAV, Mexico*; P. Rico, J. Gómez Ribelles,* Valencia Polytechnic University, Spain*; M. Pacha Olivenza, M. Gonzales Martin ,* University of Extremadura, Spain

In the present study, multi-layer coatings of hydroxyapatite (HA) - Ag/TiO2-TiN/Ti were obtained on the Ti-6Al-4V alloy, by means of the magnetron sputtering technique. During its evaluation, the techniques of energy dispersive X-ray spectroscopy, X-ray diffraction (DRX), field emission scanning electron microscopy, transmission electron microscopy, micro scratch test, nano indentation, were used. The biological response was evaluated by means of I) cytotoxicity and adhesion of mouse mesenchymal stem cells, and II) adhesion and bacterial viability of Staphylococcus aureus strain. HA coatings with a Ca/P from 1.67 to 1.76 were obtained, whose structure was verified by means of DRX and Micro-Raman spectroscopy. Coatings evidenced a multi-layer architecture. A decrease in hardness and an increase in adhesion to the substrate of the coatings were obtained by incorporating the intermediate layers. The biological evaluation carried out indicated an antibacterial effect and potentially non-toxic character in the coatings.

Tribology and Mechanical Behavior of Coatings and Engineered Surfaces
Room Golden State Ballroom - Session EP-ThP

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

EP-ThP-2 Atmospheric Pressure Plasma Deposition of Low Friction Coatings on Engineering Thermoplastics: The Plasma-Process-Structure in the Versatile Spray Coating Technique as Basis of Commercial Applications*, **Dietmar Kopp (dietmar.kopp@joanneum.at)**, J. Lackner, R. Kaindl, W. Waldhauser,* Joanneum Research Forschungsgesellschaft GmbH, Austria*; M. Stummer,* INOCON, Austria*; A. Coclite,* Graz University of Technology, Austria

Deposition strategies to obtain low friction and wear resistant coatings on 3D-printed, rough polyamide 12 (PA, nylon) surfaces were developed by atmospheric pressure plasma deposition. Dry lubricants such as MoS_2 and graphite were fed as powder material to the DC plasma torch by INOCON Technologie GmbH, applying parameter sets that would not thermally damage the heat sensitive polymers. In general, microtribological characterization indicates that especially high influence on the coating performance is given by the coating composition and thickness. Pure MoS_2 coatings are quickly worn, especially on roughness tips (mean substrate roughness R_a up to 20 µm), exposing the substrate locally and leading to friction coefficients similar to PA12 and rather limited potential of wear protection. Compound coatings with graphite within a MoS_2 matrix provide huge potential for wear protection (>20 times less penetration depth) even in the case of exposing PA12 to roughness tips, due to the transfer of lubricating carbon into these exposed areas. If the thickness is sufficiently high, missing wear of tips results in low-friction behavior with friction coefficients <0.1 (thus, 4 times lower than for uncoated PA12).

EP-ThP-3 The Influence of Boron in Thick AlTiN and AlCrN Coatings Deposited by Bipolar HiPIMS to Control Residual Stress and Improve Tribomechanical Properties*, **Adrián Claver (adrian.claver@unavarra.es)**,* Institute for Advanced Materials and Mathematics (INAMAT2), Universidad Pública de Navarra (UPNA), Spain*; I. Fernandez,* Nano4Energy SL, Spain*; J. Endrino,* Nano4Energy SL, University Loyola, Sevilla (Spain), Spain*; J. Santiago ,* Nano4Energy SL, Spain*; J. Fernández Palacio,* Centre of Advanced Surface Engineering, AIN, Spain*; J. García,* Institute for Advanced Materials and Mathematics (INAMAT2), Universidad Pública de Navarra (UPNA), Spain

Thick PVD coatings provide wear protection, improving thermal and abrasion resistance during machining operations[1]. In order to improve tool life, multiple attempts have been made to increase the thickness beyond 5 um [2]. However, ion bombardment during film growth usually induces high compressive stress in thick films, resulting in adhesive failure of coated tools in tribological applications. In this work, we investigated how to reduce high stress levels in thick AlTiN- and AlCrN- based coatings [3] (up to 10 µm) by incorporating boron to the metal nitride structure. The composition and microstructure of the coatings were characterized using Glow Discharge Optical Emission Spectrometry (GDOES), Scanning electron microscopy (SEM), transmission electron microscopy (TEM) and X-ray diffraction (XRD). The stress of the coatings was determined by curvature method and compared with the results derived from XRD analysis. Nano-hardness, scratch tests and pin-on-disk tests were carried out to study the tribological properties of the coatings. Machining tests were performed to validate in service performance of the coatings. The results show that standard AlTiN and AlCrN are hard and dense coatings but highly stressed. However, the incorporation of boron induces a nanocomposite type structure, formed by nanocrystalline domains and boron-rich amorphous regions. As a result, stress is reduced, while thermal stability, elasticity and fracture toughness are enhanced.

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EP-ThP-5 Optimized a-SiC_x:H Intermediate Layers for Well-adhered a-C:H Thin Films on Ferrous Alloys*, V. Piroli, J. Weber, M. Goldbeck,* UCS, Brazil*; F. Cemin,* UNICAMP, Brazil*; A. Michels,* UCS, Brazil*; F. Alvarez,* UNICAMP, Brazil*; **Carlos Figueroa (cafiguer@ucs.br)**,* UCS, Brazil

Hydrogenated amorphous carbon (a-C:H) thin films have been gaining much attention among new technologies due to their low friction coefficients and high wear resistance. However, the low adhesion of the couple a-C:H thin film / ferrous alloy is still an ongoing challenge. The use of Si-containing interlayers not only minimizes residual stress and provides chemical affinity but also these intermediate layers can be obtained by cheap PECVD technologies. Nevertheless, high temperatures in the interlayer's deposition process, e.g., 300 °C [1], do not allow massive applications due to the degradation of previous substrate's heat treatment. This work aims to study the adhesion of a-C:H thin films on AISI 4140 steel, with an a-SiC_x:H interlayer, by using relatively low a-SiC_x:H deposition temperatures to enable the scale production of well-adhered a-C:H/a-SiC_x:H/steel structures.

Samples of AISI 4140 steel were treated in a low-cost pulsed-DC PECVD equipment with electrostatic confinement. The a-SiC_x:H interlayer was deposited for 10 min using a tetramethylsilane (TMS) / Ar vapor mixture. During the interlayer's deposition, the temperature varied between 85 °C and 200 °C, according to the process. The a-C:H film was deposited using an acetylene (C_2H_2) / Ar gas mixture for 60 min at 80 °C.

The thin films were characterized by FEG-SEM, optical microscopy, XPS and scratch test. Cross-section FEG-SEM allowed observing three well-defined regions in the sandwich structure: the a-C:H film (top layer), the a-SiC_x:H film (intermediate layer) and the steel (bottom layer). One can observe an exponential decay of the defects density by increasing the interlayer's deposition temperature. XPS determined the content of oxygen present in the a-C:H/a-SiC_x:H interface, which varied between 1.5% (for 85 °C) and 0.5% (for 200 °C). The critical scratch loads (Lc), obtained from the scratch test, varied from 4.3 N (for 85 °C) to 5.9 N (for 175 °C). To date, samples with Si-containing interlayers produced from hexamethyldisiloxane (HMDSO), TMS or tetraethoxysilane (TEOS) at 500 °C presented maximum a-C:H critical loads of 3.4 N, 0.4 N and 0.3 N, respectively [2]. Finally, the use of relatively low temperatures on the deposition of well-adhered, with minimum oxygen content a-C:H/a-SiC_x:H/steel structures represents a new finding. The results of this research could facilitate industrial production of coatings with a more affordable market price.

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EP-ThP-6 A Deep Neural Network for Pattern Optimization of Microtextured Surfaces in Lubricated Contacts*, A. Silva, **Veniero Lenzi (veniero.lenzi@fisica.uminho.pt)**, L. Marques,* University of Minho, Portugal

The reduction of friction between moving parts in contact in mechanical equipment is fundamental in order to reduce wear, increase equipment efficiency and reduce the use of lubricant. It is well-known that the treatment of surfaces through micro-texturing allows for an improvement of the tribological performance of the contact, however it is not clear how to find the optimal texturing pattern that provides the best friction reduction.

In this work, we designed a deep neural network (DNN) that can predict the Stribeck curve of a lubricated contact, starting solely from the knowledge of the initial texture. The DNN has been trained using a in-house developed finite elements method solver for the Reynolds equation that correctly treats the cavitation problem by using the iterative inexact Newton algorithm. The training set for the DNN consisted of randomly generated patterns of dimples with fixed depth and radius.

Our DNN is capable of calculating Stribeck curves in milliseconds with a mean square error lower than 0.5 % when compared to curves obtained with the direct solver. This enabled us to solve the inverse problem, that is finding the optimal texturing given a Stribeck curve, by means of a search within the solution space obtained using the DNN.

EP-ThP-7 e-Poster Presentation: Tribological Behavior of Lamellar Solid Lubricant Coatings in Low Viscosity Hydrocarbons*, Euan Cairns* ***(euancairns@my.unt.edu)****, A. Ayyagari,* University of North Texas, USA*; S. Berkebile,* US DEVCOM Army Research laboratory, USA*; D. Berman, S. Aouadi, A. Voevodin,* University of North Texas, USA

Transitioning to multi-fuel capability of modern combustion engines has generated need for new sources of effective lubrication inside fuel pump assemblies. Low-viscosity fuels pose a tribological challenge when used in fuel injection systems, as they provide lower lubricity when compared to conventional diesel fuels, leading to degradation of service life of fuel pump and injection system components. Lamellar solid lubricants (MoS_2, WS_2, graphite, h-BN) may become a good secondary source of lubrication when applied to such components as coatings. However, their tribological behavior in fuel environments is unknown. In this report, we compared sliding wear performance of lamellar solid lubricant coatings applied by burnishing onto the surface of yttria-stabilized zirconia (YSZ) coating and tested in ethanol and dodecane environments. The results showed that the tribological behavior of the solid lubricants depends on their composition and environment. MoS_2-based coatings provide the best performance when compared to hexagonal BN, graphite, and WS_2, when tested in a high-frequency reciprocating sliding against steel counterpart. The coefficient of friction of MoS_2 in dodecane is 0.08 for the first 5,000 cycles, while for other solid lubricants it is above 0.1. Possible lubrication mechanisms are discussed, where interactions between fuel molecules and the lamellar lubricant structure as well as oxidations processes may affect the lubrication mechanisms. The discussions of these mechanisms are supported with SEM/EDS and Raman spectroscopy analyses of the tribological contacts

EP-ThP-10 Mechanical Behaviour and Effects of Cu/Ni Nanolaminate Coatings on the Fatigue Properties of Welded Steel Specimen*, Jakob Brunow* ***(jakob.brunow@tuhh.de)****, M. Rutner,* Hamburg University of Technology, Institute for Metal and Composite Structures, Germany

Nanostructured metal multilayers have been known to possess exceptional material properties regarding strength, magnetism, radiation resistance, fatigue resistance, corrosion resistance and abrasion resistance. Since there are a lot of governing factors for the material properties of nanostructured metal multilayers (NMMs), i.e. global composition, local composition, used metals and interface strength, NMMs can be tailored to specific needs. [1]

Cu/Ni NMMs have seen special interest in the scientific community for the wide variety of possible applications and the possibility to synthesize with a single-bath electrodeposition process. [2]

Current research at the University of Hamburg is focusing on the strengthening of welded steel structures against fatigue, by coating the surface with a nanostructured Cu/Ni-metal laminate patch. [3] The findings are in accordance with other studies that studied the fatigue properties of Cu/Ni nanolaminate coatings. [4]

The surface roughness is measured with AFM before and after fatigue loading. The nanolaminate Cu/Ni coating on the fractured fatigue samples are examined with serial FIB cross sectioning. Subsequently a TEM-lamella is taken from the crack tip to investigate the crack initiation and assess the fracture mechanisms of the Cu/Ni NMM. The Cu-layers experience multi-crack formation and the initial multi-cracks are arrested at the interfaces. The Ni layers bridge those cracks and each layer ruptures individually, resulting in a distinctive crack pattern.
The lifetime enhancement of the welded sample are linked to the fracture mechanism of the NMM and can thus be attributed to the following five mechanisms: reduced surface roughness, micro-crack bridging, energy dissipation, crack arrest at interfaces and changes in surface tribology. [5]

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EP-ThP-11 Nanoindentation Spectrometry*, Esteban Broitman* ***(esteban.daniel.broitman@skf.com)****,* SKF B.V., Netherlands

The precise knowledge of material microstructures is of vital importance to understand their mechanical and tribological performance. Standard microstructural characterization, carried out by optical and electron microscopy together with X-ray diffraction, is usually correlated to the hardness determined by Rockwell or Vickers indentation at macro- and microscale, and nanoindentation at nanoscale.

In the first part of the presentation, the background of a novel statistical nanoindentation technique to measure hardness and Young's modulus of materials is described [1]. We indicate how experiments are designed, and how the distribution of the hardness and modulus of elasticity determined from the nanoindentation observations are deconvoluted to generate hardness histograms that reflect unique characteristics (fingerprints) of each coating or bulk material. We show how the statistical deconvolution analyses gives an estimate of the microstructural constituents, their volume fraction and corresponding plastic and elastic properties at nanoscale. In the second part, numerous examples on different kind of coatings and bulk materials are presented to illustrate the usefulness of the novel technique.

We demonstrate that, by using nanoindentation as a novel tool for static nanomechanical spectrometric analysis of coatings and bulk materials, a fundamental understanding of the relation between local microstructure (phases and their size) and local material response during elastic and plastic deformation can be obtained.

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