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-1 e-Poster Presentation: Combinatorial Study of Mo_2N-Cu Coatings to Optimize Tribological Performance in Low Viscosity Fuel Environments, ***Slater Caldwell***, *M. Dockins, E. Cairns,* University of North Texas, USA*; S. Berkebile,* US DEVCOM Army Research Laboratory, USA*; A. Voevodin, D. Berman, S. Aouadi,* University of North Texas, USA

Wear-induced failure between sliding mechanical interfaces causes a reduction in component lifespan and subsequently a system failure. These problems are exacerbated when the environment is insufficiently lubricated, such as in the case of low viscosity fuels used in automotive and aerospace engines. In the current study, a combinatorial and high throughput magnetron sputtering method was used to determine the optimum copper content in Mo_2--N-Cu coatings that provides the best tribological response. Mo_2--N-Cu is a tribo-catalytically composite whereby copper promotes the formation of a carbon-based lubricious film at sliding interfaces. More specifically, metal nitride films with copper inclusions provide both strength and wear resistance whereby copper clusters act as catalysts for carbon film formation when paired with synthetic oils. Hence the goal of this study is to understand the interplay between the catalytic activity of copper that improves the lubricity of the interface and the mechanical properties that diminish for high copper and to optimize such properties to minimize wear. Therefore, Mo_2--N-Cu coatings were produced by reactive magnetron sputtering from Mo and Cu targets in a controlled nitrogen environment. The structural and chemical properties of these materials were evaluated using X-ray diffraction (XRD) and an environmental scanning electron microscope (ESEM) equipped with energy dispersive X-ray spectroscopy (EDS). The mechanical and tribo-catalytic properties were tested using a nanoindenter and a high frequency reciprocating rig (HFRR) tribometer in decane.

EP-ThP-2 Triboactive CrAlXN Coatings for Wear and Friction Reduction under Grease Lubrication, *K. Bobzin,* Surface Engineering Institute - RWTH Aachen University, Germany*; C. Kalscheuer,* surface Engineering Institute - RWTH Aachen University, Germany*;* ***Max Philip Möbius***, Surface Engineering Institute - RWTH Aachen University, Germany*; M. Rank,* Institute of Machine Elements, Gears and Tribology - TU Kaiserslautern, Germany*; M. Oehler, O. Koch,* Institute for Machine Elements, Gears and Tribology, Germany

The continuous development of tribological systems to increase their energy efficiency and sustainability is of increasing social and economic interest. The chain drive as a common machine element offers open potential in this respect. The service life of a chain is determined by its wear-related elongation. The wear of chain pins in the chain joint can be reduced by CrAlN coatings. This component also leads to most frictional losses in a chain drive. When lubricating with greases, friction- and wear-reducing reaction layers are formed in interaction with the steel surface. Interactions of CrAlN coatings with the lubricating greases, however, are limited due to their chemically inert properties. Triboactive CrAlMoN coatings offer a promising solution. In this study CrAlMoN coatings deposited by magnetron sputtering physical vapor deposition (MS-PVD) were tribologically investigated via pin-on-disc tribometer (PoD) and compared to a CrAlN coating and an uncoated reference. All systems were tested with two fully additivated grease references and two basic greases, which exclusively contain a S- or P-additive. To approach the application, selected combinations were then tested in a chain-joint tribometer (CJT), a component test bench for chain drives. The coatings were successfully deposited on chain pins. The incorporation of the triboactive element Mo leads to a lower indentation hardness H_{IT} of the coatings. The CrALMoN coating with a high Mo content achieved a low total wear volume and lead to a stable and relatively low coefficient of friction (CoF) in contrast to the other tested coatings. This coating leads to low levels of friction and total wear in combination with a basic grease +S equivalent to a steel surface in combination with a fully additivated grease lubricant. The results show the high potential of triboactive coatings for wear and friction reduction in chain drives. Additionally, expensive and environmentally dangerous anti-wear and extreme pressure additives can be avoided using the triboactive CrAlMoN coating.

EP-ThP-3 Influence of Nb and Ta Added Elements on the Corrosion and Mechanical Properties of CrYN Coatings, ***İhsan Efeoğlu***, *B. Yaylalı, G. Gülten, Y. Totik,* Atatürk University, Turkey*; P. Kelly, J. Malecka,* Manchester Metropolitan University, U.K.

Barrier coatings are applied to many machine elements that are exposed to aggressive/hardservice conditions to prevent corrosion, oxidation and wear at high temperatures. These coatings are widely used to protect structural components of gas/steam turbines in the energy and aerospace industries against aggressive operating conditions. Looking at the usage areas in general terms, it is seen that thermal barrier coatings (TBC) are of vital importance in terms of environmental and socio-economic aspects. With the increase in technological developments, gas/steam turbines and high temperature components such as aircraft/jet engines are expected to operate under supercritical conditions and with minimum losses. In this study, Nb and Ta doped CrYN film was coated on 316L stainless steel (SS) using CFUBMS (Closed Field Unbalanced MagnetronSputtering) technique. Then, the corrosion resistance, structural and mechanical properties of CrYN:Nb/V solid thin films were investigated. Microhardness device was used for mechanical properties. SEM and XRD techniques were used to study the microstructural characteristics of the coatings on the silicon wafer. Corrosion properties were investigated using potentiostat test unit NaCl solution. The highest hardness was found as 38.60 GPa for CrYN:Nb film and 26.47 GPa for CrYN:Ta. The results show that the coated samples have higher corrosion resistance than the uncoated samples. In addition, it has been observed that the corrosion resistance of Ta doped CrYN thin films is relatively better than Nb doped thin films.

EP-ThP-4 Evaluation of the Adhesive Strength of a Nitrided Stainless Steel Under Cyclic Contact Loads, *D. Fernández-Valdés,* ***Jesús Vidal-Torres***, SEPI ESIME Instituto Politécnico Nacional, Mexico*; A. López-Liévano,* Universidad Veracruzana, Mexico*; G. Rodríguez-Castro, A. Meneses-Amador,* SEPI ESIME Instituto Politécnico Nacional, Mexico

In this study, the adhesion resistance of a nitrided AISI 316L stainless steel is analyzed by standing contact fatigue. The AISI 316L steel was nitrided by the salt baths process where three nitride layers were obtained. Hardness and Young's modulus values were obtained by nanoindentation testing. The H^3/E^2 ratio was used as an indicator of fracture resistance of the nitride layers. Fracture toughness of the nitride layers was achieved by scratch testing. The cyclic contact loads were applied to nitrided steel by means of an alumina spherical indenter. The layer/substrate systems were evaluated under 50,000 load cycles and maintaining a constant frequency of 5 Hz. The Hertzian stress state caused by the contact loads was obtained using the finite element method. Two different damage zones were observed, one at the periphery of the contact zone, where cohesive damage was caused by the maximum principal stresses and the other inside the contact zone where adhesive damage was caused by the maximum shear stress. The adhesive resistance of the nitride layers was a function of both layer thickness and H^3/E^2 ratio of each system.

EP-ThP-5 Effect of Annealing Treatment on Mechanical Properties of Nanostructured Metallic Films Deposited by Pulsed Laser Deposition, ***Francesco Bignoli***, CNRS, France*; S. Rashid, E. Rossi,* Università degli studi Roma 3, Italy*; P. Djemia,* CNRS, France*; M. Sebastiani,* Università degli studi Roma 3, Italy*; A. Li Bassi*, Politecnico di Milano, Italy*; M. Ghidelli,* CNRS, France

The design of metallic thin film with controlled composition and microstructure has become increasingly important for industry applications, involving strong mechanical solicitations. Specifically, metallic glasses (MGTFs) and high entropy alloys thin films (HEATFs) have shown a great potential due to their unique combination of large mechanical properties such as yield strength (3 GPa) and ductility (10%) [1,2]. However, the relationship microstructure-mechanical properties are not fully understood since the morphological control is often limited by the most employed sputtering deposition. In this field, Pulsed Laser Deposition (PLD) offers the possibility to widely control the morphology of the films by simply changing the process parameters, affecting the growth mechanisms from atom-by-atom to cluster-assembled growth regimes. Recently, PLD has shown a large potential for the deposition ZrCu MGTFs and CoCrCuFeNi HEATFs reporting large and tunable mechanical properties such as an elastic modulus and hardness of 175 and 11 GPa [2].

Here, we explore the possibility to further nanostructuring PLD deposited ZrCu compact and nanogranular MGTFs by performing annealing treatments from 300 up to 550°C, while investigating the devitrification process and the evolution of the mechanical properties.

Structural characterization shows that compact films remain amorphous up to 420°C, while the crystallization process of nanogranular films is completed at 420°C due to the combination of high interface density, free volume and O content [3]. We show that the mechanical properties increase with the annealing temperature due to the progressive crystallization reaching a plateau upon complete crystallization with elastic modulus and hardness up to 180 and 14 GPa, respectively. Furthermore, we show that compact films have residual tensile stress from 169 to 691 MPa whose magnitude increase as a function of the temperature due to nanocrystalline phase nucleation followed by grain growth. On the other hand, nanogranular films show a maximum residual stress of 1.1 GPa at 420°C followed by a decrease at higher annealing temperatures, indicating a complete crystallization.

Overall, we show that PLD in combination with post-thermal annealing can generate different families of metallic films with varying nanoscale morphologies, resulting in tunable mechanical properties and thermal stability with potential as structural coatings.

References:
1. Y. Zou et al., Nat. Commun., 6, 7748, 2015.
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3. F. Bignoli et al., Mater. Des., 221, 110972, 2022.

EP-ThP-6 Accurate Measurement of Thin Film Elastic Properties Using Thermal Loading*, C. Trost,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences & Dept. of Materials Science, Montanuniversität Leoben, 8700 Leoben, Austria*; S. Zak,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Austria*;* ***Megan J. Cordill,*** Erich Schmid Institute of Materials Science, Austrian Academy of Sciences & Dept. of Materials Science, Montanuniversität Leoben, 8700 Leoben, Austria

Measuring the elastic modulus of novel metallic thin films, such as intermetallics or high entropy alloy (HEA) films, can be challenging. Nanoindentation is the most used method to determine the hardness, but has recently been shown that nanoindentation is not a reliable method to measure elastic modulus of films less than 5 µm thick without having an influence from the substrate [1] (especially in case of stiff thin film on compliant substrate). Therefore, another method should be utilized to accurately measure thin film elastic modulus. One approach that has not been considered is to use in-situ stress measurements while heating the film. From the initial slope of the heating and cooling stress-temperature curves, a substrate-free elastic modulus can be calculated. In order to determine if this technique is viable, known metal films of Mo and Al on silicon substrates will first be evaluated and compared to literature and nanoindentation of the same film systems. Then, more complex, metastable MoAg alloys and refractory HEA films, with applications in power electronics, will be evaluated.

[1] S. Zak, C.O.W. Trost, P. Kreiml, M.J. Cordill, J. Mater. Res. (2022).

EP-ThP-7 Microstructural, Mechanical and Tribological Properties of TiAlSiN–Cu Superhard Nanocomposite Coatings Deposited by Filtered Cathodic Arc Ion Plating Technique*,* ***In-Wook Park****, S. Heo, W. Kim, J. Kim, E. Choi, S. Choe,* Korea Institute of Industrial Technology (KITECH), Republic of Korea*; J. Lim,* BMT Co., Ltd, Republic of Korea

The effects of the Cu content on the microstructural, mechanical and tribological properties of the TiAlSiN–Cu coatings were investigated in an effort to improve the wear resistance with a good fracture toughness for cutting tool applications. A functionally graded TiAlSiN–Cu coating with various copper (Cu) contents was fabricated by a filtered cathodic arc ion plating technique using four different (Ti, $TiAl_2$, Ti_4Si, and Ti_4Cu) targets in an argon-nitrogen atmosphere. The results showed that the TiAlSiN–Cu coatings are a nanocomposite consisting of (Ti,Al)N nano-crystallites (~5 to 7 nm) embedded in an amorphous matrix, which is a mixture of TiO_x, AlO_x, SiO_x, SiN_x, and CuO_x phase. The addition of Cu atoms into the TiAlSiN coatings led to the formation of an amorphous copper oxide (CuO_x) phase in the coatings. The maximum nanohardness (H) of ~46 GPa, H/E ratio of ~0.102, and adhesion bonding strength between coating and substrate of ~60 N (L_{C2}) were obtained at a Cu content ranging from 1.02 to 2.92 at.% in the TiAlSiN–Cu coatings. The coating with the lowest friction coefficient and best wear resistance was also obtained at a Cu content of 2.92 at.%. The formation of the amorphous CuO_x phase during coating growth or sliding test played a key role as a smooth solid-lubricant layer, and reduced the average friction coefficient (~0.46) and wear rate (~10×10^{-6} $mm^3/N{\cdot}m$).

EP-ThP-8 Adhesion of WTi to Polyimide Measured by Complementary Methods*, D. Gutnik,* ***Alice Lassnig,*** Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben, Austria*; A. Kleinbichler,* Infineon Technologies AG, Austria*; P. Imrich,* KAI Kompetenzzentrum Automobil- und Industrieelektronik, Austria*; M. Cordill,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben, Austria

Adhesion of metal films to polymers is an increasing area of research. Not only are metal-polymer interfaces found in flexible and foldable electronics, but also in rigid microelectronics. While several methods to quantify the metal-polymer interface adhesion are available, a direct comparison of the available methods on the same interface has not been performed yet. In this work, the adhesion of the WTi-Polyimide (PI) interface was evaluated with methods, using spontaneous buckles and the tensile induced delamination model. One unique aspect of the samples is that all fabrication processes were performed at the wafer level and testing performed on released WTi-PI dogbone shaped tension samples. After being released from the rigid wafer, spontaneous cracking and buckling occurred due to the release of residual stresses in the WTi film. The results show that both models lead to similar adhesion energies. The similarity of adhesion results indicate that both methods are suitable to assess interface strength and that the two methods can be confidently compared.

EP-ThP-9 On the Use of Integrated 3D-Profilometry to Bring New Insights on Mechanical Characterization of PVD Coatings by Scratch and Tribology Tests*,* ***Philippe Kempe,*** Rtec-Instruments SA, Switzerland

Coatings bring advantages for functional surfaces on different relevant mechanical performances: higher wear resistance for a longer lifetime or lower friction coefficient for a reduced energy consumption. The physical phenomena involved at the surfaces under stress conditions represent a key part in understanding the performances of some coatings. Scratch testing characterizes the scratch resistance and adhesion of coatings. Tribology testing characterizes the wear resistance and friction of surfaces in relative contacts. The unique combination of mechanical testing with 3D-profilometry (confocal microscopy and white-light interferometry) provides new insights into the visualization of surface damages occurring in scratch testing (cracks, plastic deformation and delamination of coatings) and tribology (modes of wear and deformation). Examples of characterization with 3D imaging on PVD coatings are shown.

EP-ThP-10 Typical Gaffes During the Nanoindentation of Coatings*,* ***Esteban Broitman,*** SKF - Research and Technology Development, Netherlands

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 easily done by just *"the pushing of one button."* However, the consequences of easy procedures have led many researchers to multiple publications with erroneous data.

Recently, we have reviewed the nanoindentation hardness of materials at macro, micro, and nanoscale (E. Broitman, Tribology Letters, vol. 65, 2017, p. 23). Some misconceptions in the nanoindentation technique were highlighted, and solutions to errors were proposed. In this paper, five typical 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 wrong area selection to calculate instrumented indentation hardness; (2) the wrong data conversion from Vickers microindentation to Berkovich nanoindentation; (3) the confusion of thermal drift with creep and viscoelastic effects; (4) the wrong correlation of hardness with tip penetration; (5) the preconceptions about a direct relationship between elastic modulus and hardness.

The origins of the aforementioned mistakes will be elucidated from the lack of understanding on contacts mechanics theory, the limits and validation of the Oliver and Pharr's method, and preconceptions transmitted from generation to generation of nanoindenter 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 coatings.

EP-ThP-11 High Temperature Tribological Behavior of TiAlN Coatings with Different Ti/Al Ratios*, C. Pereira, F. Amorim,* Pontifícia Universidade Católica do Paraná, Brazil*; G. Souza,* UEPG, Brazil*; P. Soares, M. Meruvia,* ***Ricardo Torres,*** Pontifícia Universidade Católica do Paraná, Brazil

Dry machining is an advanced process that places higher demands on machine tools, as the temperature at the interface between a cutting tool edge and a metallic workpiece can vary from 200°C to over 1,300°C. This requires the tool material to have extremely high hardness and thermal toughness besides good wear and adhesion resistance. The use of coatings which act as coolant, isolating the tool from the heat of the cut, improve tool performance and increase the tool lifespan. TiAlN, a currently used coating with high hardness at high temperatures and good wear resistance, has properties that depend greatly on its crystal structure, which are Al content and annealing temperature dependent. Thermal annealing at 700-900°C causes decomposition of $Ti_{1-x}Al_xN$ solid solution into stable c- or w-TiN and AlN, which affect the coating microstructure and, consequently, its properties. Therefore, this work aims to investigate the effect of the Ti/Al ratio on the tribological behavior of $Ti_{1-x}Al_xN$ coatings when submitted to wear conditions at 20 °C, 500 °C and 800 °C. For that samples of Futura Nano® TiAlN ($Ti_{0,56}Al_{0,44}N$) and the Latuma® AlTiN ($Ti_{0,37}Al_{0,63}N$) coatings, deposited on tungsten carbide (WC) hard metal discs by cathodic arc physical vapor deposition (CAE/PVD) by Oerlikon Balzers Revestimentos Metálicos LTDA, were used. The coatings were physico-chemically analyzed by energy dispersive spectroscopy (EDS), X-ray diffraction (XRD), X-ray photoelectron (XPS) and Raman spectroscopy; their hardness and elastic modulus were determined, the adhesion evaluated and the tribological performance investigated at 20 °C, 500 °C and 800 °C temperatures performing pin-on-disk wear testing. The results show that for both coating, the annealing up to 800 °C gradually induces the solid solution to decompose in c-TiN and AlN, followed by formation of TiO_2 and Al_2O_3, that lead to decrease in hardness, more pronounced in TiAlN 800 °C (35%) than in AlTiN (18%), and in elastic modulus. COF in the same trend show a more pronounced increase in COF for TiAlN than for AlTiN when the temperature is raised up to 500 °C, followed by a sharp decrease in COF with increasing temperature. For both coatings the wear mechanism changes from abrasive to oxidative with temperature raise and, while no change in failure mechanism is observed in TiAlN, that buckles under compression, for AlTiN it changes from delamination at 20 °C to spalling at 800 °C.

EP-ThP-12 Structural, Phase, Electrochemical, and Tribological Evaluation of TiO_2 and SiO_2 Multilayer Coatings Obtained by Reactive Magnetron Sputtering with Potential Biomedical Applications*,* ***Julián Andrés Lenis Rodas,*** University of Antioquia, Politécnico Colombiano Jaime Isaza Cadavid and Servicio Nacional de Aprendizaje - SENA, Colombia

In the present study, TiO2 and SiO2 multilayer coatings were obtained by reactive magnetron sputtering using Ti and Si targets in the presence of a mixture of Argon and oxygen. The cross-sectional structure of the coatings was evaluated by scanning electron microscopy, while the chemical composition was determined by energy dispersive X-ray spectroscopy. The phases present in the coatings were analyzed by micro Raman spectroscopy and X-ray diffraction. Additionally, corrosion resistance tests were performed using electrochemical impedance and polarization resistance techniques. Finally, the wear resistance of the coatings was evaluated using a pin-on-disk tribometer.

EP-ThP-13 Tribological Performance of Sliding Pads Against Ti_6Al_4V for Aeronautic Applications*,* ***Manel Rodriguez Ripoll****, A. Ventura,* AC2T Research GmbH, Austria

The extension and retraction of flaps in commercial aircrafts relies on the displacement of rolling elements along a flap trap made of titanium alloy. A typical flap support relies on 2 sets of 4 rollers to transfer flight/ground and lateral loads from the flap panel into the wingbox. These rollers are part of the carriage assembly and need to be re-greased every C-check (~7500 flight cycles).

With the aim of extending the maintenance intervals of rollers, the present work evaluates the possibility of replacing the sets of rollers by sliding pads. To this end, prospective sliding pad materials are evaluated under reciprocating sliding against Ti6Al4V plates using a flat-on-flat configuration. The experiments are performed under contact conditions similar to those encountered by the rollers in actual flap configurations.

The selected materials comprise a self-lubricating nickel base coating, a high-performance cupper nickel alloy and a diamond-like carbon coating. After the experiments, the total wear volume on the titanium plate is measured using 3D microscopy. Both, the pin and the plate are investigated using scanning electron microscopy in order to elucidate the dominating wear mechanism. Based on the results obtained, the best performing candidates will be upscale via component tests.

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