

Tribology and Mechanical Behavior of Coatings and Engineered Surfaces

Room Town & Country B - Session E3-1-TuM

Tribology of Coatings and Surfaces for Industrial Applications I

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

8:00am **E3-1-TuM-1 Carbon Based Coatings Deposited Over Aisi 4140 to Improve Wear Resistance in Machine Components**, *F. Delfin*, UTN, Argentina; *D. Heim*, Upper University of Applied Sciences, Wels Campus, Austria; *Sonia Brühl*, UTN, Argentina

INVITED

Carbon based coatings or DLC are well known to have a low friction coefficient, chemical stability at room or low temperatures, high hardness and also corrosion resistance. Three different carbon-based coatings were deposited over plasma nitrided or non nitrided AISI 4140 mild steel to improve hardness and wear resistance so as tribological properties.

One of them is an amorphous hydrogenated thick Si doped DLC (a-C:H:Si) deposited by PACVD, in a Rübigen facility in Austria using a DC pulsed discharge, with acetylene and HMDSO as carbon and silicon precursors, respectively. It will be compared with a Si-free DLC (a-C:H) deposited in the same conditions. The third one is a multilayer film with DLC as top coating deposited by the PVD PEMS technique in a Cemecon facility in Argentina. In this case, the anchor layer consists of CrN and the top layer is a chromium-doped hydrogenated amorphous carbon (a-C:H:Cr).

Full characterization was carried out by SEM, Raman Spectroscopy and XRD. Mechanical properties were obtained using nanoindentation. Tribological behavior was studied with Pin-on-Disk tests, evaluating wear loss and friction coefficient, and complementary SEM/EDS analysis on the wear track and on the counterpart. Adhesion was assessed with variable load Scratch Test and Rockwell C indentation method. Abrasion tests following ASTM G65 standard were also conducted.

All coatings contain hydrogen and a combination of sp^3 - sp^2 bonding, with high disorder level caused mainly by the dopants. Low coefficients of friction were obtained with the films, between 0.1 and 0.3, being the lowest the Si-free thick DLC reaching 0.06. Regarding the wear rates with alumina as counterpart, only the Si-free DLC and the duplex nitrided steel plus a-C:H:Cr presented a low volume loss. Adhesion was also improved by the plasma nitriding pre-treatment in this coating, which provided a hardness gradient in the substrate to support the load applied. Meanwhile, Si doping improved DLC wear resistance in the case of abrasion tests.

As a conclusion, the thick and Si-free DLC coating has the lowest CoF and wear rate when deposited over mild steel. On the other hand, the thin PVD Cr doped DLC coating presented the best performance when deposited over nitrided steel. This also improved the adhesion comparing to the other two PACVD coatings. The Si-doped DLC failed in the pin-on-disk test because of the adhesion with the counterpart, but it presented a good wear resistance in abrasion. Tribological mechanisms, and applications of each type of coating will be discussed.

8:40am **E3-1-TuM-3 Tribological Behaviour of Diamond Coated Reaction-Bonded Silicon Carbide Under Dry and Seawater Environment**, *R. Kannan*, N. C. Indian Institute of Technology Madras, India; *R. Ganguly*, *S. Mandal*, *S. Rao*, Carborundum Universal Limited, Industrial Ceramic Division, India; *M.S Ramachandra Rao*, Indian Institute of Technology Madras, India

Ceramic seal materials such as silicon carbide (SiC) and Reaction-Bonded SiC (89%SiC and 11% free Silicon) are widely used in mechanical pumps, compressors, and feed water pumps to prevent the fluid leakage between the two rotating shafts. In a continuously harsh environment, these mechanical seal materials undergo high friction and wear due to the high asperity-to-asperity contact between the two mating parts and generate high frictional heat at the interface. This scenario induces seal failure under high operating conditions. Especially in the corrosive medium such as acids, high pH value solutions and hard abrasive media can cause aggressive seal failure. To reduce production downtime and improve the longevity of the mechanical seal, the surface of the seal material needs to be engineered with a suitable coating that will satisfy the required properties of high hardness, chemically inertness, and low friction coefficient. In this regard, we have come up with a super hard coating material such as diamond

(Micro-crystalline) on the surface of Reaction-Bonded SiC using the hot filament CVD (HFCVD) method to meet the above-required properties. In this work, we have carried out a systematic study on the tribological effect of uncoated and diamond-coated seal material against silicon nitride ceramic under a dry and seawater environment. The initial physical characteristics such as surface morphology, surface roughness, and Raman analysis were performed on the samples before and after coating. To understand the friction and wear characteristics, the linear reciprocating tribometer test (Ball on Flat) was conducted on the uncoated (18,000 cycles) and diamond-coated (36,000 cycles) samples using a universal tribometer. The experimental results show that the diamond-coated sample exhibits a low friction coefficient (0.05 ± 0.01) compared to the uncoated sample (Friction coefficient (CoF) = 0.24 ± 0.037) at a normal load of 10 N with a frequency of 10 Hz under a seawater environment. Similarly, under dry conditions for the same tribo-test parameters, the diamond-coated sample (CoF = 0.06 ± 0.02) outperforms compared with the uncoated sample (CoF = 0.42 ± 0.17) at a percentage improvement of 85%. From the observed results, we conclude that diamond-coated ceramic seals are considered a suitable alternative to commercially available seals in terms of high operating conditions and durability under both wet (Sea water) and dry running operation. All these results will be presented.

9:00am **E3-1-TuM-4 Friend or Foe? The Role of Oxygen in the Tribological Performance of Solid Lubricant MoS₂**, *Andrey Bondarev*, *I. Ponomarev*, *T. Polcar*, Czech Technical University in Prague, Czechia

MoS₂ is a solid lubricant used in various forms, such as a dry lubricant by itself, or as a component of a more complex coating. In both these forms, the effect of oxygen contamination on the sliding properties of MoS₂ coatings is traditionally considered detrimental, resulting in expensive technological processes to produce pure MoS₂.

The interaction of the film with oxygen can be split into three phases. First, during magnetron sputtering, oxygen is incorporated into the growing film from the residual atmosphere. Then, storing the film in the open air can cause oxidation. Finally, the coatings experience a negative influence of molecular or atomic oxygen from the surrounding atmosphere on their tribological properties. Despite the origin, a formation of a noticeable amount of molybdenum oxides on the sliding surface has been reported as the reason behind the poorer tribological properties.

The Mo-S-O coatings were fabricated by unbalanced magnetron sputtering. It was found that hardness, elastic modulus, and wear resistance can all be enhanced with the addition of oxygen. The Mo-S-O coatings were tribologically tested under vacuum, ambient air, and nitrogen. A high oxygen content, up to 38 at.%, does not prevent the formation of a lubricious crystalline MoS₂ tribofilm, which is formed through triboactivated segregation of the deposited Mo-S-O amorphous coatings into a crystalline MoS₂-based phase and an amorphous S-depleted Mo-S-O phase. By means of advanced TEM and EELS studies, it was shown that an ultra-thin crystalline MoS₂ tribolayer incorporates some amount of oxygen. Such an imperfect tribolayer was found to reduce the coefficient of friction to 0.02, a value lower than that of pure MoS₂ under vacuum (0.05). Molecular dynamics simulations performed using a newly developed Mo-S-O force field confirmed that such an imperfect tribolayer can mitigate friction in a manner comparable to MoS₂. Results of DFT simulations support the experimental findings and show that partial substitution of S atoms to O atoms in the MoS₂ crystal structure does not increase shear modulus. Tribological tests of the Mo-S-O coatings after 1-year storage under ambient air (RH = $35 \pm 5\%$) revealed the same trends as freshly deposited samples – the Mo-S-O coatings outperform pure MoS₂ coatings demonstrating lower values of friction coefficient.

In this work it is shown that contradictory to common knowledge, adding oxygen to a solid lubricant MoS₂ coating could be beneficial. Since eliminating oxygen in MoS₂-based solid lubricants is extremely costly, our findings can significantly reduce the production and storage cost for aerospace and other vacuum applications.

9:20am **E3-1-TuM-5 Friction and Wear Behavior of Electrostatically Developed Nanocomposite Coatings for High Performance Cutting Tool**, *S. Ramanujam*, *Rakesh Kumar Gunda*, *K. Ramanjaneyulu*, *K. Sudhakar Reddy*, *K. Ankamma*, *A. Ramchandra Reddy*, Mahatma Gandhi Institute of Technology, India

In modern industry, mechanical parts are subjected to friction and wear, leading to heat generation, which affects the reliability, life and power consumption of machinery. Numerous attempts have been made in the past to reduce the amount of friction created during manufacturing operations by implementing strategic approaches like enhancing the

Tuesday Morning, May 23, 2023

quantity of lubricating fluid and the practice of employing materials possessing high hardness and abrasion resistance. Although cutting fluids are employed to control the high machining zone temperatures, their usage possesses health hazards and in addition leading to higher machining cost. One of the most effective ways to reduce friction and wear is to coat surfaces in contact with abrasion-resistant materials. Several coating techniques have been developed over time for a variety of uses. The current investigation deals with studies concentrated on the usage of hard coatings in order to reduce friction coefficient for improving the friction and wear performance.

High-voltage electrostatic coating technique has been developed with an aim to improve nanocomposite coating on tungsten carbide tool and to eliminate the cutting fluid usage in machining process. Sliding experiments were performed on pin-on-disc tribometer to examine the friction and wear behavior of electrostatic coated nanocomposite pin material under dry sliding condition at various sliding speed and applied loads. To understand the wear characteristics of sliding coated pin, scanning electron microscopy and optical microscopy is used to examine. The obtained results observed that electrostatic coated pin performed tremendous improvement in tribological properties compared to uncoated WC pin material. The present work findings offer new insights into typical nanocomposite hard coatings on cutting tools exhibits remarkable characteristics beneficial to diverse industrial applications.

Keywords: Hard coatings, nanocomposite, electrostatic technique, friction, wear

9:40am E3-1-TuM-6 Modification of Diamond Like Carbon (DLC) to Improve Specific Tribological Characteristics for Automotive Applications, Denis Romagnoli, F. Lavalle, STS srl, Italy

INVITED

Carbon-based materials play an important role in today's science and technology. Carbon is a very versatile element whose two most interesting allotropic forms are Diamond (sp³) and Graphite (sp²). In recent years, there have been continuous and important advances in carbon science, such as the chemical vapor deposition of diamond, the discovery of fullerenes, carbon nanotubes, and the single layer of graphene. At the same time, the DLC has consolidated all its applications in the automotive, mechanical in general but also fashion sectors. An amorphous carbon layer can have different ratios between diamond C and graphitic C. The management of these fractions but above all the architecture of the layer generates different types of DLC that allow to enhance the hardness, contain friction and improve the corrosion resistance of the component.

The demand for the application of DLC on components for endothermic and hybrid engines (pins, valves, camshafts) is constantly growing; about the novelties in the field of mobility, the need to protect the gears of electric motors from wear or corrosion the bipolar plates of hydrogen propellers are increasingly pressing. DLC can increase surface hardness, reduce friction and protect against corrosion; for racing this means an improvement in terms of overall engine performance, and for the automotive sector an improvement in yield with the consequent possibility of reducing emissions into the atmosphere, allowing the use of certain components in non-standard conditions (e.g. rotation constantly at a maximum number of revolutions in an electric motor) and use different materials (for example steel compared to graphite in a Fuel Cell). The possibility therefore of being able to manage hardness, coefficient of friction, and corrosion resistance in the same DLC layer is determined for the application.

In this presentation, the DLC deposited with hybrid technology PVD-PaCVD (Physical Vapor Deposition- Plasma assisted Chemical Vapor Deposition) will be examined. The modification of the layers, the thicknesses, the compositions, and the containment of surface defects allow enhancing the characteristics of hardness, reduction of friction, and resistance to corrosion of the layer. These characteristics can therefore be objectified with laboratory instruments such as nanoindenter, contact tribometer, and salt spray chamber. The next step is the bench test to then move on to the final validation and production launch.

10:20am E3-1-TuM-8 Fabrication and Tribological Behaviors of DLC Coatings Embedded with Graphene Nanoplatelets, Guizhi Wu, R. Brittain, A. Morina, University of Leeds, UK; E. Broitman, SKF Research & Technology Development Center, Netherlands; L. Yang, University of Leeds, UK

Graphene has garnered widely interests in tribology due to its nature of reducing friction. The lamellar structure endows graphene a weak shearing stress, and thus a low friction. Utilizing graphene platelets to wrap around nanodiamond particles to form nanoscrolls at a sliding interface would

decrease coefficient of friction (COF) significantly by achieving an incommensurate contact on diamond-like carbon (DLC) coatings. However, such complicated design containing too many additives will reduce the stability of the system. COF of TiN ceramic film will decrease by simply dripping down a graphene aqueous dispersion on the sliding interface, by which a smooth sliding interface promoted by graphene are formed. Nevertheless, the most concern is that graphene might be easily pushed out of the friction interface at the beginning of sliding, thus may not affect the friction test. Graphene nanocrystallites can also be fabricated by vapor deposition method and embedded in the carbon film matrix to form a nanocomposite carbon film, of which the COF decreases significantly. But it remains to be further demonstrated whether the synthesized carbon crystallites is graphene.

In this study we report the synthesis and tribological behaviors of DLC coatings embedded with Graphene nanoplatelets (GNP), namely DLC-GNP nanocomposites. GNP layer was deposited by two methods, namely, spin-coating and spray-coating. The DLC layer was deposited on top of the GNP coated coupon by plasma enhanced chemical vapour deposition. The effect of GNP on the lubricated tribological response under elevated temperature was discussed. The results show that GNP has a tendency to agglomerate for higher GNP coverages by using spin-coating, which would lead to removal of the GNP more easily and negate the friction reduction effect of the GNP. The tribotests show decrease in friction and wear of the DLC-GNP nanocomposites in various GNP coverages, with the lowest friction (COF ~0.03) and wear (~1.6 x 10⁻¹⁹ m³/Nm⁻¹) achieved, which might be due to a highly graphitic transfer film featuring low shearing stress formed in the friction interlayer. This study might contribute to energy conservation and emission reduction, and also promote the development of green tribology under the global 'Carbon net zero' aim.

10:40am E3-1-TuM-9 Tribological Properties MoS₂-WC Duplex Coatings in Low Viscosity Hydrocarbons, Euan Cairns, University of North Texas, USA; S. Dixit, Plasma Technology Inc., USA; D. Berman, S. Aouadi, A. Voevodin, University of North Texas, USA

Emerging applications using low-carbon emission fuels, such as ethanol and dodecane, lead to a growing need for lubricious materials that can improve the tribological behavior and increase the wear life of fuel pump components. In our previous studies we found that MoS₂ spray-coat on 52100 steel had the potential to be used in low-viscosity hydrocarbon fuels as a protective coating. The best results were observed in dodecane fuel, where a coefficient of friction of less than 0.1 was maintained for 100 m of sliding. In this report, we extend the understanding of MoS₂ spray coating performance in low viscosity hydrocarbons to a duplex coating with a hard, wear-resistant underlayer made of coated and ground finished High Velocity Oxygen Fuel (HVOF) thermal spray tungsten carbide (WC) combined with the lubricious MoS₂ top layer to create synergistic tribological protection of 52100 steel substrates from the sliding wear. Tribological tests were performed under the reciprocating sliding condition in dodecane and ethanol, followed by investigations with scanning electron microscopy equipped with an energy dispersive spectrometer, Raman microscopy, and x-ray photoelectron spectroscopy for microstructural and compositional analysis. The effects of substrate roughness and composition on the wear life of the coatings were examined using optical profilometry and microscopy of wear tracks. The results were used to identify lubrication and sliding wear protection mechanisms when using MoS₂-WC duplex coatings prepared by spray methods.

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**, Universidad de Chile, **Manel Rodriguez Ripoll**, AC2T Research GmbH, Austria

1:40pm **E1-1-TuA-1 Chemistry and Mechanical Properties of 2D Transition Metal Carbides and Carbonitrides (MXenes)**, **Vadym Mochalin**, University of Missouri S&T, USA **INVITED**

A large family of two-dimensional transition metal carbides and nitrides (MXenes) raises interest for many applications due to their high electrical conductivity, mechanical properties [1], potentially tunable electronic structure [2], nonlinear optical properties [3], and the ability to be manufactured in the thin film state [4]. However, their chemistry that is key to development of these applications, still remains poorly understood [5-8]. In this presentation we will discuss recent progress in understanding fundamental MXene chemistry and harnessing it for suppressing unwanted reactions and prolonging stability of these materials.

Mechanical properties of MXenes, including their adhesion to other materials, 2D materials, and other MXenes will be discussed. Adhesion plays an important role in assembly of 2D heterostructures. Data on tribological properties of MXenes, including superlubricity, will also be presented and discussed.

Selected examples illustrating connections between MXene chemistry and their mechanical properties will also be considered.

References

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2:20pm **E1-1-TuA-3 Tribocorrosion Behaviours of VNbMoTaWCr High Entropy Alloy Coatings**, **Ismail Rahmadtulloh**, C. Wang, W. Wang, National Taiwan University of Science and Technology, Taiwan; B. Lou, Chang Gung University, Taiwan; J. Lee, Ming Chi University of Technology, Taiwan

Recently, tribocorrosion has become an interesting research topic for academic researchers and industries. The deep understanding of tribocorrosion behaviour has become important in development of alloys having good wear resistance and corrosion protection. In this work, four VNbMoTaWCr high entropy alloy (HEA) coatings with different Cr contents were fabricated by a pulsed DC magnetron sputtering system. For tribocorrosion tests, the HEA coatings were immersed in a 3.5 wt.% NaCl aqueous solution at room temperature under 1 N load using a pin-on-disk tribometer. The potentiodynamic polarization tests were performed to investigate the corrosion potential, corrosion current density, and polarization resistance for each coating before and during the tribocorrosion test. The effect of Cr concentrations on the tribocorrosion behaviours of four VNbMoTaWCr HEA coatings was explored. The synergism between wear and corrosion was studied based on ASTM G119 standard. The change of corrosion loss due to wear and the change of wear loss due to corrosion were calculated. The total material loss rate and corrosion-wear map were explored to understand the extent of the wear and corrosion augmentation factors. We can find out that the VNbMoTaWCr coating with 11.7 at.% Cr content had the lowest total material loss rate of 9.2162 mm/yr, which is better than that of 304SS,

18.0009 mm/yr. It showed that the transitions between wear and corrosion for four VNbMoTaWCr coatings and 304SS were all lower than 0.1 indicating synergistic effects dominated and corrosion was affecting wear to a great extent than wear was affecting corrosion.

2:40pm **E1-1-TuA-4 Fundamentals of Phototribology**, B. Perotti, UCS, Brazil; A. Cammarata, Czech Technical University in Prague, Czech Republic; F. Cemin, Nantes Université, France; S. Sales de Mello, Université Grenoble Alpes, CNRS, France; L. Leidens, UCS, Brazil; F. Echeverrigaray, UNICAMP, Brazil; T. Minea, Université Paris-Saclay, France; F. Alvarez, UNICAMP, Brazil; A. Michels, UCS, Brazil; T. Polcar, University of Southampton, UK; **Carlos Figueroa**, UCS, Brazil

Friction phenomenon is a ubiquitous manifestation of nature originated in the dissipation of energy after stochastic interactions of particles from two surfaces. There are several ways to set up the friction behavior of tribological systems by means of surface finishing, liquids and gases, all of them nonreversible processes. Thus, the active control of friction through external sources is a challenge in tribology. Electric and magnetic fields were proposed to control friction remotely. It is a new paradigm in tribology where radiation fields can tune friction. In our case, we used a photoactive material (TiO₂) to active control of friction forces at the nanoscale as a function of the presence or absence of UV illumination ($\lambda = 365$ nm and variable luminous flux) by friction force microscopy (FFM). We could determine that the light can tune friction forces in a reversible, stable, reproducible and reliable way. The UV light induces surface rearrangements of atoms in a similar way of those processes for degradation of organic molecules on activated TiO₂ surfaces. Indeed, the half-life of the pseudo-first order kinetics is roughly the same of those well established degradation processes of organic molecules in aqueous suspension with TiO₂ particles. Moreover, the reduction of friction under UV illumination follows a sigmoidal behavior with the luminous flux. 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.

3:00pm **E1-1-TuA-5 Ultra-thin nanotwinned (CoCrNi)_{100-x}W_x Medium Entropy Alloy Film: Role of Nanotwin in Mechanical and Tribology Behaviors**, **Jhen-De You**, National Taiwan University, Taiwan; P. Yiu, Ming Chi University of Technology, Taiwan; C. Hsueh, National Taiwan University, Taiwan

A significant amount of materials are lost each year as a result of wear-induced damages. Meanwhile, small-scale medium entropy alloy films (MEAFs) have attracted much attention recently due to their superior mechanical properties over their bulk counterparts. In this work, we aim to investigate the plastic deformation characteristics (hardness, strain rate sensitivity and tribology) and structure evolution of nanotwinned (CoCrNi)_{100-x}W_x ($x = 0, 0.9, 3, 5.3, 7.2$ and 9.8) MEAFs. The (CoCrNi)_{100-x}W_x MEAFs were fabricated using magnetron co-sputtering. The tungsten content increased from 0 to 22.01 at.% with the increasing power applied on the W target. Microstructures of thin films thus prepared were examined by the atomic force microscope, scanning electron microscope and transmission electron microscope. The structure evolution and mechanical properties were investigated in this work. The W addition in CoCrNi matrix resulted in transition from FCC solid solution to amorphous structure. Nanotwins were observed in FCC grains for $x \leq 9.8$, and the W_{7.2} MEAF showed the thinnest twin thickness of ~ 1 nm, the superior wear-resistance and hardness, and the lower coefficient of friction via nano-scratch, nano-wear and nano-indentation tests. Although the amorphous W_{22.01} MEAF showed the higher hardness than W_{7.2}, its wear resistance was inferior to crystalline W_{7.2} MEAF. Our work demonstrated the structure evolution, mechanical properties, and wear performance of (CoCrNi)_{100-x}W_x MEAFs, also gave insight of wear-mechanism in nanotwinned CoCrNi-based system.

4:00pm **E1-1-TuA-8 Understanding the Tribology Behavior of Carbon Thin Films Deposited by the HiPIMS Technique in Ar+Ne Atmospheres**, **Cesar D. Rivera Tello**, Universidad de Guadalajara CUCEI, Departamento de Ingeniería mecánica eléctrica, Mexico; L. Flores Cova, A. Guerrero de León, J. Pérez Alvarez, M. Flores Martínez, Universidad de Guadalajara CUCEI, Mexico

This investigation focusses in the effects of using Ne + Ar mixture in the plasma of the HiPIMS discharges in the deposition process of diamond like carbon thin films and in the tribological tests. The plasma deposition process was analyzed by mass quadrupole spectroscopy obtaining the ion energy distribution for C⁺ ions from different gas composition discharges

(20, 40, 60, 80, 90 and 100% Ne). Derived from these processes, a carbon thin film was obtained for each gas composition discharge, where the bond-structures of them were analyzed by Raman spectroscopy. The tribology behavior of all the samples was studied by friction coefficients and analyzing the images of the wear track obtained by reciprocating ball-on-plate configuration of the tribological tests. Furthermore, these Ne +Ar mixture thin film were compared to similar films without Ne to observe mainly differences. The results showed slight increment on the ionization with the %Ne gas composition discharge. Besides, a significant reduction of wear in comparison to the films without Ne was shown, derived from lower sp^3 content in the carbon films in comparison to the carbon films without Ne. Finally, we observe a significant increment on the deposition rate for the films deposited with Ne.

4:20pm E1-1-TuA-9 Mechanical and Tribological Behavior of Cr-Y-N Multilayers Grown by Hipims as a Function of Progressive Changes in the Coating Architecture, *R. Chefer Apolinario, P. Tavares Avila, J. Nascimento Pereira*, São Carlos School of Engineering (EESC), University of São Paulo (USP), Brazil; *P. Daum*, Fraunhofer Institute for Mechanics of Materials IWM, Germany; *C. Greiner*, Institute for Applied Materials (IAM), Karlsruhe Institute of Technology (KIT), Germany; *Haroldo Cavalcanti Pinto*, São Carlos School of Engineering (EESC), University of São Paulo (USP), Brazil

One of the most promising physical methods for coating deposition is the High-Power Impulse Magnetron Sputtering (HiPIMS) technology, a variation of magnetron sputtering that allows for denser and harder films due to the application of high-intensity power pulses with very short duration in the range of a few microseconds to increase plasma density. Hardness, fracture toughness, tribological and corrosion behavior of the coatings can be further designed using nanostructures. In the present work, the coating architecture of Cr-Y-N multilayers was systematically modified to provide superior hardness and wear resistance. To this end, a novel methodology that introduces a progressive substrate movement in front of the target during sputtering with alternating nitrogen flow is proposed. Misorientation gradients were superimposed to the multilayers by growing with alternating nitrogen flow rates between 50 and 10 sccm. The misorientation gradients were generated using progressive periods of oscillation: none, ± 5 , ± 10 , ± 15 , ± 20 and ± 25 degrees in front of a CrY target. Coating morphology, preferential orientation of grain growth and the distributions of nitrogen and yttrium across the multilayers are discussed based on scanning electron microscopy, transmission electron microscopy, X-ray diffractometry, residual stress analyses, X-ray photoelectron spectroscopy, instrumented nanoindentation wear resistance and corrosion

4:40pm E1-1-TuA-10 The Effect of Core Crystallographic Orientation on the Dislocation Dynamics of Core-Shell Nanostructures: A Molecular Dynamics Study, *Robert Fleming*, Arkansas State University, USA

Core-shell nanostructures, composed of a metallic core with a hard amorphous shell, are known to exhibit unusual dislocation dynamics which subjected to compression loading. Experimentally, this behavior manifests as substantial deformation recovery beyond the elastic limit, enhanced fatigue resistance, and improved durability. However, the fundamental physical mechanisms that enable the underlying dislocation dynamics in these nanostructures are still poorly understood. In this study, the role of crystallographic orientation of the confined metallic core is investigated for 2 FCC metals (Al, Cu) and 1 HCP metal (Mg), all with a-Si shells. Along with supporting stress calculations, understanding the interplay between crystallographic orientation, active slip systems, and core-shell interface structures will provide insights into the unique mechanical behavior of these nanostructures, with an ultimate goal to design material systems with controllable dislocation dynamics.

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, Michael Chandross, Sandia National Laboratories, USA, Andreas Rosenkranz, Universidad de Chile

8:20am E1-2-WeM-2 Coatings and Composites for Extreme Environments Lab (CE)2, Achyuth Kulkarni, T. Ansell, Naval Postgraduate School, USA

The Aluminum (6xxx.x) series alloys have gained widespread use across several industries, including aerospace, marine, structural, and automobile, primarily due to their impressive characteristics, such as low density, high specific strength, formability, and good corrosion resistance. However, despite these advantages, these alloys are susceptible to poor mechanical strength and wear resistance when exposed to challenging environmental conditions. To tackle these shortcomings, the development of Aluminum Metal Matrix Composites (MMC) has emerged as a crucial advancement in improving the properties of commercial Al alloys without compromising the ductility of their substrate material. The use of MMCs offers the potential to enhance the mechanical properties of Al alloys significantly, including tensile strength, hardness, wear resistance, and fatigue strength, thereby expanding their application range in harsh environments. Moreover, the incorporation of various reinforcing materials, such as ceramic particles, whiskers, fibers, and nanotubes, into the Al matrix can further augment its mechanical properties.

This study presents the synthesis of novel nanocomposite aluminum-based coatings reinforced with boron carbide (B_4C) microparticles and Graphene Nanoplatelets (GNPs) using a dual-reinforcement approach. The combination of the two reinforcing materials activates synergistic interactions between particles, resulting in improved dispersion and enhanced strengthening efficacy. To evaluate the efficiency of the dual-reinforcement approach, coatings containing 4 vol.% of GNPs and 4 vol.% B_4C are compared with unreinforced Al coatings and coatings reinforced with either 4 vol.% BNNT or 4 vol.% B_4C . The mechanical properties of the coatings are evaluated using adhesion testing, and nanoindentation. The structural characterization is done using Optical Microscopy, and Field Emission Scanning Electron Microscope (FE-SEM). Moreover, the elevated temperature (300°C) dry sliding wear characteristics are studied. The worn surfaces are further analyzed using FESEM and a 3-D non-contact type optical profilometer. These findings have significant implications for the development of high-performance nanocomposite coatings in various industries, including aerospace, automotive, and structural applications.

8:40am E1-2-WeM-3 Tribological Behavior of MoS_2 Based Coatings Under Different Sliding to Rolling Lubricated Contact Conditions, Newton Fukumasu, I. Machado, University of São Paulo, Brazil; R. Rego, Aeronautics Institute of Technology, Brazil; A. Tschiptschin, R. Souza, University of São Paulo, Brazil

Powertrain components, such as gears and bearings, are used in a variety of sectors, including automotive, aerospace, energy conversion and manufacturing. These parts are subjected to high torque, cyclic loading and variable sliding to rolling ratio (SRR), which can induce sliding wear and/or rolling contact fatigue damage. Strategies to mitigate those failures include the use of advanced 2D coating materials, such as molybdenum disulfide, which may exhibit excellent solid lubrication properties under high contact stresses and pure sliding conditions. In principle, higher SRR conditions would demand coatings with higher hardness to mitigate sliding wear while lower SRR conditions would require more ductile coatings to improve RCF life. Given those demands, adaptive coatings that can change their properties in response to local contact conditions would improve component performance and durability. One mechanism to provide such local adaption is the shear-induced crystal orientation (SCO) promoted by high contact sliding condition, in which the highly oriented stresses and elevated local temperatures may lead to the formation of nanocrystalline regions on the surface of the material, resulting in improved mechanical properties, including increased hardness, fracture toughness and/or lower friction. In this work, MoS_2 based coatings were deposited by pulsed direct current magnetron sputtering technique and submitted to 1 GPa contact pressure under variable SRR (10% and 100%) to evaluate conditions more prone to promote the SCO mechanism. SAE 52100 steel discs were coated and tested in a ball-on-disc configuration using a Mini Traction Machine

(PCS Inc.) equipment, in which both disc and ball tangential velocities were controlled to provide different SRR levels, maintaining a low average velocity to provide a boundary lubrication condition. Numerical simulations of tested configuration, using the Finite Element Method, indicated no significant correlation between shear stress developed at contact region and SRR values, while shear stress peak values were obtained for higher coefficient of friction. Experimental results indicated elevated coefficient of friction in the running in part of the test for all SRR conditions, decreasing to a lower stable COF along the test. The lower COF was achieved for highest SRR condition, in which Raman spectroscopy indicated the existence of a crystalline MoS_2 phase inside the wear track, not present initially in the as-deposited condition. These results support the SCO mechanism, adapting local coating properties that may improve wear and RCF resistance.

9:00am E1-2-WeM-4 Catalytic Transformation of Lubricants to Wear-Protective Tribofilms on Selected Steel Surfaces During Sliding, Yip-Wah Chung, A. Khan, J. Ahmed, T. Martin, S. Liu, Northwestern University, USA; S. Berkebile, Army Research Laboratory, USA; Q. Wang, Northwestern University, USA

We report here the impact of different alloying elements in steels on friction and wear behavior by performing ball-on-flat lubricated reciprocating tribotesting experiments on 52100 ball on steel flats with different compositions heat-treated to give similar hardness and microstructure, with polyalphaolefin (PAO-4) and/or *n*-dodecane as the lubricant. The major observation is that steels containing high concentrations (≥ 5 wt.%) of Cr, Mo, V, or Cu/Ni give rise to markedly reduced wear compared with 52100 or plain carbon steels, with D2 steel, which contains 11.5 wt.% Cr, as the major alloying element being the most wear-resistant. The wear resistance is strongly correlated with the efficiency of formation of carbon-containing oligomeric films at surfaces as determined by Raman spectroscopy. Given the strong affinity of chromium to oxygen, chromium should exist as Cr_2O_3 at the steel surfaces during testing. We have performed molecular dynamics simulation on Cr_2O_3 and demonstrated its ability to catalyze the formation of carbon-containing oligomeric films from hydrocarbon molecules, consistent with its known catalytic activity in other hydrocarbon reactions. We believe that chromium-containing alloys, such as D2, and coatings, such as CrN, derive their wear resistance in part from the efficient *in-situ* formation of wear-protective carbon tribofilms at contacting asperities.

9:20am E1-2-WeM-5 Aromatic Compounds as Sustainable Lubricants for Iron, Sophie Loehlé, TotalEnergies, France INVITED

The present work will focus on one type of aromatic compound, Hypericin, as a nature constituent of St. John's wort, which is commonly known as anti-depressant, anti-virus, and anti-biotic agents. Besides its functions in medical treatment, the present work reveals its potential as an original friction modifier. Indeed, amazing tribological properties were observed by adding hypericin to glycerol in order to lubricate a steel/SiC tribopair. A hypericin concentration as low as 0.3% reduces friction coefficient (CoF) from ~ 0.02 to a value below 0.01 (the so-called superlubricity regime) under boundary lubrication. The excellent friction reduction property of hypericin derives from its hydroxygen functionalized carboxypolycyclic structure, which achieves surface protection by forming a nanometer-thick hypericin-like tribofilm on both steel and SiC surfaces. The tribologically-induced polymerization of the molecules into graphene is detected by high resolution transmission electron microscopy (HRTEM) on a focused ion beam cross-section. First-principles calculations elucidate the thermodynamic driving force for the process, monitored in real time by *ab initio* and classical, molecular dynamics simulations. This work suggests an exciting and unconventional way to promote the formation of graphene by mechanical stresses and uncovers the great potential of many aromatic molecules derived from the pharmacopoeia, such as hypericin, as lubricants for industrial applications.

11:00am E1-2-WeM-10 How Efficient Is the Self Adaption Concept for Low Friction with TMD-Based Sputtered Coatings, Albano Cavaleiro, University of Coimbra, Portugal INVITED

In last decades a huge amount of research was dedicated for further improvement of the frictional behaviour of sputtered coatings based on transition metal dichalcogenides (TMD). It is currently accepted that the low friction achieved with this type of coatings is based on the development of a tribolayer in the contact formed by a self-adaption process. The sliding process gives rise to the establishment of contact conditions which are able, by a reorientation / recrystallization process, to transform the material in the contact leading to the formation of a

Wednesday Morning, May 24, 2023

tribolayer with the required low friction characteristics. Whatever are the initial structure and phase composition in the sputtered deposited coatings, the transformation can occur in the contact if specific conditions of sliding exist.

In this talk, the conditions for activating the self adaption process will be presented and discussed. Sliding and contact parameters such as, the number of cycles, the temperature and the contact pressure, will be related with the type of TMD-based sputtered coatings and antagonist materials with the supporting of several practical examples. Tribological testing, using different experimental rigs, and detailed characterization of the materials in the contact are in the basis of the interpretation of the results to be presented.

11:40am E1-2-WeM-12 Self-Lubricating Titanium Alloys: Design and High Temperature Tribological Performance Up to 800 °C, *H. Torres, K. Pichelbauer, S. Budnyk*, AC2T Research GmbH, Austria; *T. Schachinger, C. Gachot*, TU Wien, Austria; **Manel Rodriguez Ripoll**, AC2T Research GmbH, Austria

Titanium and its alloys are extensively used in aerospace and biomedical applications thanks to their high strength-to-weight ratios, excellent corrosion resistance or good biocompatibility. However, a general widespread of these alloys to other applications, such as machine elements is severely limited by their poor tribological properties.

With the aim of improving the performance of titanium alloys and spreading their applicability to new fields, the present work deals with the development of self-lubricating titanium alloys, with an emphasis on high temperature applications performance. The alloy developed is deposited by means of laser melting using a direct diode laser system, as laser deposition processes can be readily used for additive manufacturing techniques that offer a great flexibility and efficiency compared to traditional subtractive manufacturing processes.

A mixture of Bi with Ni powder (70:30) is pre-placed on a Ti6Al4V plate and melted with the direct diode laser. The melting process results in the presence of a 300 µm thickness coating on top of the Ti6Al4V plate. The microstructure and phase composition of the deposited self-lubricating titanium alloy coating is characterized using X-ray diffraction, scanning and transmission electron microscopy. The results reveal the presence of three distinct phases, a Ti6Al4V matrix with Bi-Ti-rich inclusions that are surrounded by a Ni-Ti-rich phase. Afterwards, the friction and wear performance of the developed self-lubricating coating is evaluated using high temperature tribological tests at temperatures ranging from room temperature up to 800 °C. The tribological experiments are performed against Ti6Al4V pins in reciprocating sliding.

The results reveal that the self-lubricating laser deposited titanium alloy is able to significantly decrease friction in the 400 to 800°C range, when compared to conventional Ti6Al4V. In terms of wear, the resulting self-lubricating alloy was also able to clearly outperform the reference Ti6Al4V in all the evaluated temperature ranges, from RT to 800°C. The friction reduction mechanism in the 400 to 600°C temperature range has been linked to the transfer and smearing of bismuth-rich phases to the counter body. At the highest temperature of 800°C, friction reduction could be linked to the formation of the ternary oxide BiVO₄, which could act as an effective solid lubricant. This overall tribological performance makes the presented self-lubricating alloy a potential candidate for numerous high temperature applications in the aerospace and energy sectors.

12:00pm E1-2-WeM-13 Electrodeposited of Silver Nano-Particules Plant Based to Improve Lubrication of Composite Films, *Pierre-Antoine Gay*, Haute Ecole Arc Ingénierie, Switzerland; *I. Markovic Milosevic*, HEPIA Institut inSTI, Switzerland; *T. Journot*, HE ARC Ingénierie, Switzerland; *J. Maurer*, Faculty of Biology and Medicine. Clinical pharmacology, Switzerland

Electrodeposition of silver and nanoparticles plant based like TiO₂ and Al₂O₃ has been successfully demonstrated for the first time in silver electrodeposit composite films. Their tribological properties and wear resistance were investigated by a pin-on-disk type friction testing using an electrical system to also measure the resistivity of the composite coatings.

The friction coefficient of silver/TiO₂ composite films decreased with increasing TiO₂ content. Plating parameters, nanoparticles concentration and zeta potential were systematically investigated in order to found a relationship between incorporation rate *V_p* and friction coefficient. Ag – TiO₂ with 60g/L of nanoparticles composite film showed the minimum friction coefficient value, with wear resistance 40% better. A comparison

with electrodeposit composit coatings was discussed with traditional nanoparticles.

Wednesday Afternoon, May 24, 2023

Tribology and Mechanical Behavior of Coatings and Engineered Surfaces

Room Pacific F-G - Session E3-2-WeA

Tribology of Coatings and Surfaces for Industrial Applications II

Moderators: **Nazlim Bagcivan**, Schaeffler Technologies GmbH & Co. KG, Germany, **Rainer Cremer**, KCS Europe GmbH, Germany, **Philipp Grützmaier**, Institute of Engineering Design and Product Development, Austria

4:20pm **E3-2-WeA-8 Hard MoSC Solid Lubricant Coating**, **Tomas Polcar**, *I. Ponomarev*, *T. Vitu*, Czech Technical University in Prague, Czech Republic

Doping of transition metal dichalcogenides with carbon results in coating with much better mechanical properties (adhesion, hardness) than that of pure MoS₂. Solid lubrication is achieved through tribochemical reaction during sliding, which produces a thin MoS₂ tribolayer with basal planes parallel to the coating surface. The coating is a superb lubricant in a vacuum and provides a very low coefficient of friction (lower than typical DLC) in the humid air. However, MoSC prepared by d.c. sputtering is still a relatively soft coating with a hardness of 2-4 GPa, and chemical composition (e.g. carbon content) cannot be further optimized due to an insufficient understanding of tribolayer formation.

We deposited amorphous MoSC coating by co-sputtering MoS₂ and C targets in HIPIMS mode, leading to enhanced hardness close to 8 GPa and improved adhesion on steel substrates. Special attention has been paid to tribological testing in various environments (vacuum, humid air) and a formation of a low-friction MoS₂ tribolayer. Experimental results are compared with molecular dynamics simulations of sliding thanks to our recently developed force fields for Mo-S-C material combinations. It seems that the sliding induces the segregation of the carbon phase facilitating the formation of a solid lubricant tribolayer.

4:40pm **E3-2-WeA-9 Coating Solutions for Wind Turbine Bearings**, **Esteban Broitman**, SKF - Research and Technology Development, Netherlands; *T. von Schleinitz*, SKF GmbH, Germany

Wind power, a clean and affordable alternative to fossil-fuel power generation, is becoming one of the most preferable choices around the world thanks to novel technical and engineer advances, and also costs drops.

The operational challenges for wind turbines are enormous, like the permanent demand to increase turbine power and size, the work in extreme weather conditions, increasing heavy loads, need of installation in remote locations, etc. In turn, significant advances in rolling bearing designs, materials and heat treatments engineering, and coating technologies have helped to achieve enhanced performance, reliability, and increased service life at many parts of the wind turbines.

In this presentation, we will introduce three kind of coatings used at different bearing locations of the wind turbine: aluminum oxide INSOCOAT®, SKF tribological black oxide, and carbon-based NoWear®. The deposition, mechanical, tribological and electrical properties of the coatings will be discussed. Results will illustrate how far the use of coated bearings have evolved in meeting operational demands and improving the productivity and profitability of wind turbines.

5:00pm **E3-2-WeA-10 Scratch Testing and Tribology Combined with Integrated 3D-Profilometry for in-Depth Characterization of Damage Modes in PVD Coatings**, **Philippe KEMPE**, Rtec-Instruments SA, Switzerland
Scratch testing has been used to characterize the adhesion of PVD and CVD coatings. It has been normalized to different standards (ISO 20502 and ASTM C1624) which are in much use now in different industries.

The principles of the method are based on a extremely high stress at the interface between coating and substrate which generates strong damages and potential delamination in the material structure. For the development of new coatings, the correlation with real situations is not necessary straightforward and is discussed.

The integration of a 3D profilometer (confocal microscopy and white-light interferometry) combined with mechanical testing (scratch tests and tribology) allows to visualize different damages in the coating structure. It can be used with advantages in R&D projects. Different modes of testing

with scratch tester are explained and could foresee various automated modes for quality control.

5:20pm **E3-2-WeA-11 Structural, Electrochemical, and Tribological Evaluation of Silver - Hydroxyapatite Multilayer Coatings Obtained by Magnetron Sputtering with Potential Application in Implants**, **Julián Andrés Lenis Rodas**, University of Antioquia, Politécnic Colombiano Jaime Isaza Cadavid and Servicio Nacional de Aprendizaje - SENA, Colombia

In the present study, silver-hydroxyapatite multilayer coatings were obtained by magnetron sputtering. The structure of the coatings was evaluated by scanning electron microscopy and transmission electron microscopy. Chemical composition and phases were determined by energy-dispersive X-ray spectroscopy, micro Raman spectroscopy, and X-ray diffraction. The corrosion resistance was evaluated using electrochemical impedance and polarization techniques, while the wear resistance of the coatings was evaluated using a pin-on-disk tribometer.

Tribology and Mechanical Behavior of Coatings and Engineered Surfaces

Room Town & Country B - Session E2-1-ThM

Mechanical Properties and Adhesion I

Moderators: Jazmin Duarte, MPI für Eisenforschung GMBH, Germany, Alice Lassnig, Austrian Academy of Sciences, Austria, Bo-Shiuan Li, National Sun-Yat Sen University, Taiwan

8:00am **E2-1-ThM-1 Residual Stress and Interfaces in Optical Coatings for Space Applications**, *Chelsea Appleget, K. Folgner, V. Jiao, S. Dunscombe, S. Sitzman*, The Aerospace Corporation, USA; *D. White, A. Hodge*, University of Southern California, USA; *J. Barrie*, The Aerospace Corporation, USA

INVITED

Optical coatings are an enabling technology in the space industry for a variety of applications requiring high reflectance, transmittance, spectral selectivity, or thermal control. Traditional multilayered optical coatings can be designed to achieve virtually any reflectance or transmittance characteristics by tuning layer thickness, material selection, and layer material properties. For space applications, additional multifunctional performance requirements must be considered, including, but not limited to, low residual stress, good durability and adhesion in extreme environments, and tuning of surface structure. The space environment brings increasing demands on the performance of optical coatings and materials, and as such, requires an understanding of the mechanisms leading to maximized multifunctional properties.

In this talk, a discussion on adhesion, stress, and interfaces for optical films for space applications will be presented, with two case studies for residual stress and mechanical properties to follow. The first study will explore multifunctional properties of antireflection (AR) coatings and evaluation of the structure-property relationship in the context of interface character and layer microstructure in crystalline/amorphous and amorphous/amorphous multilayers. Characterization by spectrophotometry, transmission electron microscopy, and nanoindentation showed substantial variations in microstructure and film properties with tuning of layer configurations for improved transmittance. The second case study presents microstructural tailoring in highly reflective, mirrored films for improved surface scatter and residual stress performance. Thick ($>1 \mu\text{m}$) optical coatings with favorable optical properties are desired in space to protect sensitive substrates, such as radiation sensitive glass. However, traditional high reflectivity optical films, such as Ag, exhibit a degradation in optical properties due to increases in surface roughness with increasing film thickness. To circumvent these traditional limitations, the role of alloying, microstructural progression, and in-situ and ex-situ residual stress evolution is explored.

In summary, the design and fabrication of multifunctional optical coatings for space requires insight into the role of residual stresses, interfaces, delamination behavior, and microstructure, all of which inform the process-structure-property relationship in optical films.

8:40am **E2-1-ThM-3 Increased Adhesion of Mo Films on Polyimide Through Interface Modification**, *Megan Cordill, P. Kreiml*, Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Austria; *M. Rausch, C. Mitterer*, Dept. of Materials Science, Montanuniversität Leoben, Austria

Flexible and wearable electronics use metal films on polymer substrates. Here, the adhesion energy of these films to the substrates determines device performance. The interface between film and substrate is critical to adhesion energy. In this work, tensile induced delamination is used to quantify the adhesion energy of various Mo-based films on Polyimide, a common electrode material in thin film transistor displays. The method uses uniaxial straining to cause fracture of the film perpendicular and film delamination parallel to the tensile loading. Three different approaches to modify the interface to alter the adhesion energy will be compared, namely (i) the use of metallic interlayers between Mo and Polyimide (Al, Ta), (ii) the use of alloy interlayers between Mo and Polyimide (MoAl, MoTa), (iii) and the use of an alloyed Mo film (MoAl, MoTa) without an interlayer. Experiments on the different samples were performed with in-situ resistance measurements, confocal laser scanning microscopy (CLSM), and X-ray diffraction (XRD). CLSM allows for the quantification of crack density and delamination dimensions necessary for the adhesion measurement, while XRD provides film stress evolution to understand fracture and delamination mechanisms. The main aspects presented will be the

adhesion energy along with the crack and stress evolution under uniaxial tensile loading. Results show that only the use of metallic interlayers between Mo and Polyimide (Al, Ta) provide a difference in the crack and delamination densities as well as a significant increase in the calculated adhesion energy compared to any of the other modification strategies.

9:00am **E2-1-ThM-4 Picosecond Acoustics as a Local and Quantitative Adhesion Technique**, *Arnaud DEVOS, A. Vital-Juarez*, IEMN, France; *J. Desmarres*, CNES, France

The study of the bonding at interfaces between thin layers is of crucial importance in numerous applications whether they are related to optics, electronics or space exploration. A large number of methods have been developed to characterize the adhesion of a thin film to a substrate. But most of them are non local and destructive.

Acoustic waves are also sensitive to adhesion defects as they affect the way acoustic waves are reflected at the concerned interface. To address thin-films and complex stacks, ultra-high frequency acoustic waves (typ. greater than 10 GHz) are needed. Such a range is only reachable to femtosecond laser techniques known as picosecond acoustics following the pioneer work of H. Maris in the 1980's. Recently we reported several studies that show how useful can be the picosecond acoustic technique to detect and quantify adhesion defects [1][2][3].

In this paper, we prepare dedicated samples to demonstrate the local character of the adhesion measurement by picosecond acoustics. For that a fingerprint is deposited between a silicon substrate and a thin metal film. A fingerprint acts as a large adhesion defect since the wafer surface is not clean at that place when the metal film is then deposited.

We then use the picosecond acoustic technique to map the reflection coefficient of ultrahigh frequency acoustic waves along the sample surface. A clear difference is first seen on and out the zone where the fingerprint has been deposited. Second, a high resolution mapping reveals the complete shape of the buried fingerprint: bumps and hollows of the fingerprint are easily discriminated acoustically as such details do not affect the adhesion in the same manner. The acoustic map reveals the fingerprint image and the guilty person can be identified !

References:

- [1] A. Devos and P. Emery, "Thin-film adhesion characterization by Colored Picosecond Acoustics", *Surface and Coatings Technology* 352, 406 (2018).
- [2] A. Devos, A. Vital-Juarez, A. Chargui and M. Cordill, « Thin-film Adhesion: A Comparative Study Between Colored Picosecond Acoustics and Spontaneous Buckles Analysis », *Surface and Coatings Technology*, 421, 127485 (2021).
- [3] A. Vital-Juarez, L. Roffi, J.-M. Desmarres and A. Devos, « Picosecond Acoustics Versus Scotch Tape Adhesion Test: Confrontation on a Series of Similar Samples With a Variable Adhesion », 448, 128926 (2022).

9:20am **E2-1-ThM-5 What Controls Size Effects on the Mechanical Properties of Additive Manufactured Polymers**, *K. Shergill, Y. Chen, Steve Bull*, Newcastle University, UK

3D printed artefacts are becoming more common and the effect of printing parameters on their properties is key to their performance in applications. Although parameters like build orientation and raster direction are well-studied the effect of layer thickness (and its introduction of size effects into the properties of the material) is less well-known. This study determines the influence of layer thickness on the mechanical properties of polylactic acid (PLA), acrylonitrile butadiene styrene (ABS) and PETG (Polyethylene terephthalate Glycol) 3D printed specimens made with fused filament fabrication (FFF). Samples were printed with differing layer thickness and tensile tested according to ASTM D638. The study also found that when increasing the layer thickness the mechanical properties of the specimens for both ABS and PLA decreased. When it came to ultimate tensile strength, the effect of layer thickness on PLA was more significant than on ABS with PETG being in between. The differences were attributed to differences in additive layer adhesion and the effect of the structure and defects introduced by the additive layer process.

Thursday Morning, May 25, 2023

9:40am **E2-1-ThM-6 Mechanical Properties and Microstructure Evaluation of HiPIMS Cu/W and Cu/Cr bilayers with Different Thickness Ratios, Tra Anh Khoa Nguyen, T. Zhang**, Graduate Institute of Precision Engineering, National Chung Hsing University, Taiwan; *H. Wang, H. Wu*, National Chung Hsing University, Taiwan; *M. Lin*, Graduate Institute of Precision Engineering, National Chung Hsing University, Taiwan

Copper/tungsten and copper/chromium bilayer films with a total thickness of 400 nm deposited using High Power Impulse Magnetron Sputtering (HiPIMS), have been compared with those fabricated by DC Magnetron Sputtering (DCMS). The different thickness ratios of 1:3, 3:5, 1:1, 5:3, 3:1 and sputtering power effects on the surface, microstructure, morphology, the texture of sputtered films, and mechanical properties of the bilayer thin films were investigated. The results show that the HiPIMS sputtered copper/tungsten and copper/chromium bilayers exhibit higher hardness, lower electrical resistance, and lower surface roughness than films deposited under the same deposition conditions as DCMS. These properties can be attributed to the increased peak power density of the target during the HiPIMS deposition process, which increases the ion energy and enables the deposition of highly dense grain structures and changes in texture transformation. Through the results of different thickness ratios, the deposition parameter database can be used for future HiPIMS copper/ tungsten and copper/chromium multilayer deposition.

10:00am **E2-1-ThM-7 Nanoengineered Thin Film Metallic Glasses with Mutual Combination of Large Yield Strength and Ductility, F. Bignoli**, CNRS, France; *A. Brognara, J. Best*, Max-Planck Institut für Eisenforschung GmbH, Germany; *P. Djemia, D. Faurie*, CNRS, France; *A. Li Bassi*, Politecnico di Milano, Italy; *G. Dehm*, Max-Planck Institut für Eisenforschung GmbH, Germany; **Matteo Ghidelli**, CNRS, France

Thin film metallic glasses (TFMGs) are object of intense research due to their a unique combination of mechanical properties involving large yield strength (~3 GPa) and ductility (>10%) [1]. Nevertheless, the synthesis of advanced TFMGs with engineered microstructure and the understanding of their mechanical properties are barely tackled. Here, I will present recent results involving two (2) strategies to develop nanoengineered TFMGs with a controlled microstructure down to the atomic scale, resulting in outstanding and tunable mechanical properties.

In the first case, I will show the potential of Pulsed Laser Deposition (PLD) as a novel technique to synthesize nanostructured $Zr_{50}Cu_{50}$ (%at.) TFMGs. I will show how the control of PLD process parameters enables to synthesize a variety of film microstructures among which compact fully amorphous and amorphous nanogranular, showing lower density and large free volume interfaces [2]. High-resolution TEM reveals a nano-laminated self-assembled atomic structure characterized by alternated layers with different chemical enrichment [2]. This results in an unique mechanical behavior as shown by in situ TEM/SEM tensile/compression tests, reporting homogeneous deformation for nanogranular (cluster assembled) TFMGs in combination with a large yield strength (>3 GPa) and ductility (>9 %) [2].

In the second case, I will focus on the fabrication of multilayers with nanoscale period alternating either fully amorphous or amorphous/crystalline sublayers. I will show how the control of the sublayer thickness (from 100 down to 5 nm) influences the deformation behavior affecting shear bands formation, while tuning the mechanical properties. As an example, alternating CrCoNi (crystalline)/TiZrNbHf (amorphous) nanolayers results in an ultrahigh compressive yield strength (3.6 GPa) and large homogeneous deformation (~15%) [3]. Similarly, I will show the suppression of shear band/crack process in fully amorphous ($Zr_{24}Cu_{76}/Zr_{61}Cu_{39}$ %at.) multilayers with bilayer period < 50 nm, while keeping a mutual combination of large ductility (> 10%) and yields strength (>2.5 GPa).

Overall, our results pave the way to the development of novel amorphous materials with improved mechanical properties and wide application range especially in the field of microelectronics.

References:

- [1] M. Ghidelli et al. *Acta Mater.*, 131, 246, 2017.
- [2] M. Ghidelli et al. *Acta Mater.*, 213, 116955, 2021.
- [3] G. Wu et al., *Materials Today*, 51, 6, 2021.

10:20am **E2-1-ThM-8 Measurements and Simulation of Mechanical Behavior of Amorphous and Crystalline Zr(-Hf)-Cu Thin-Film Alloys, Stanislav Haviar, T. Kozák**, University of West Bohemia, Czechia; *M. Meindlhuber*, Montanuniversität Leoben, Austria; *M. Zitek*, University of West Bohemia, Czechia; *J. Keckes*, Erich Schmid Institute of Materials Science, Austria; *P. Zeman*, University of West Bohemia, Czechia

Nanoindentation and microbending testing were used to investigate the mechanical properties of Zr(-Hf)-Cu thin-film alloys prepared by nonreactive magnetron co-sputtering. A detailed analysis of nanoindentation data and microscopic images of indents allowed a more precise determination of the effective Young's modulus of the films thanks to taking the pile-up effect into account.

Microbending testing in a scanning electron microscope was performed with microcantilevers fabricated by focused ion beam and the data were evaluated using a finite element method model. As outputs of this elastoplastic model, Young's modulus, yield strength, elastic strain, apparent yield point and approximate ultimate strength and strain of the films were determined.

From a material point of view, the effect of elemental composition (Cu content and Hf substitution) and structure (glassy and crystalline) was investigated and discussed. It was shown that the substitution of Hf for Zr has less pronounced effect on the mechanical properties than the increase in the Cu content in the films that leads to a pronounced increase in the hardness, Young's modulus, elastic strain, yield strength, apparent yield point and ultimate strength but also to a decrease in the plastic parameter k and ultimate strain. Furthermore, a different atomic ordering in the crystalline and glassy Zr-Cu films of identical elemental composition results in differences in their mechanical properties and deformation behavior. The crystalline film was observed to be harder and stiffer with approximately the same elastic strain but higher yield strength and its plastic deformation was free of shear bands events.

- [1] Haviar S., Kozák T. et al. Nanoindentation and microbending analyses of glassy and crystalline Zr(-Hf)-Cu thin-film alloys. *Surf. and Coat. Technol.* 399 (2020) doi: 10.1016/j.surfcoat.2020.126139

10:40am **E2-1-ThM-9 A Nanotwinned CoCrFeNi Medium Entropy Alloy with Ultrahigh Strength Over a Wide Range of Temperature, Yun-Xuan Lin, J. Wang, C. Tsai, S. Chang, F. Ouyang**, National Tsing Hua University, Taiwan

The medium-entropy alloys (MEAs) or high-entropy alloys (HEAs) have many superior properties, including high strength, high ductility, corrosion resistance, oxidation resistance, and high temperature resistance, which can be used in many applications. In addition, nanotwin (NT) structure plays an important role on strengthening mechanism in the microscopic scale because of its special properties, such as great thermal stability, high strain rate sensitivity, low electrical resistivity, and increased mechanical strength. In this study, we combined HEA and nanotwin structure to fabricate NT-MEA thin films and investigated its mechanical properties at different temperature ranges. At first, the NT CoCrFeNi MEA films were fabricated by pulsed DC magnetron sputtering system on Si substrate. The hardness of nanotwinned CoCrFeNi medium entropy alloy films was examined by nanoindentation at varied temperatures from -80 °C to 300 °C. At the same time, the microstructures of the indentation regions were studied by transmission electron microscope (TEM). As-deposited CoCrFeNi medium entropy alloys had a columnar grain structure containing high-density nanoscale growth twins parallel to the substrate with an average twin thickness of 2.4 nm. The results show that NT-MEA was observed to have remarkably high strength, outperforming many other bulk HEAs, MEAs, nanocrystalline HEAs, and conventional NT-metals in the temperature range from -80 °C to 300 °C. The maximum hardness of NT-MEA was measured with a value of 11.3 GPa at cryogenic environment of -80°C and its hardness slightly decreased to 9.4 GPa when temperature increased from -80 °C to 300 °C. Meanwhile, neither cracks nor fractures were observed in all temperatures, suggesting that the NT-MEA was still ductile material, especially in the cryogenic environment. The

Thursday Morning, May 25, 2023

corresponding deformation mechanism at different temperature ranges would be discussed in details in this talk.

11:00am **E2-1-ThM-10 Material Properties and Mechanics of Eggshells—Nature's Survival Capsules, Jia-Yang Juang**, National Taiwan University, Taiwan **INVITED**

Amniotic eggs emerged over 300 million years ago in tetrapod vertebrates and are now the primary reproductive mode of all terrestrial amniotes. Unlike the simpler, shell-less eggs of amphibians, amniotic eggs are covered with shells, which allow the eggs to develop fully on land without drying out and thus open up new terrestrial habitats for amniotes, including birds and egg-laying reptiles. In birds, eggshells are multifunctional thin-walled structures that protect the embryo from excessive water loss, provide calcium, and sustain the weight of incubating birds. As a load-bearing structure, the eggshell must be strong enough to resist deformation and impact. Meanwhile, it must be breakable for the hatchling to emerge. Those are mechanical design trade-offs that must be adequately balanced. Here, we use combined experimental, numerical, and theoretical methods to analyze the morphology, material properties, and mechanics of 700 freshly-laid bird eggs from 58 species across three orders of magnitude in egg mass (from 1 g to 1459 g). We characterize the mineral content by acid-base titration, the crystallographic characteristics by electron backscatter diffraction (EBSD), and effective Young's modulus E by compression test and finite-element analysis (FEA). We find that the mineral content is positively correlated with E , with the lowest of 83.1% and 23.28 GPa in Zebra finch and the highest of 96.5% and 47.76 GPa in ostrich in this study. The EBSD shows that eggshell is anisotropic and non-homogeneous, with different species exhibiting different degrees of crystal orientation and texture. The experimental results are consistent with the nanoindentation test and theoretical prediction of linear elasticity. This study provides new data and insights into the material properties and mechanics of eggshells and provides clues for the future development of bioinspired multifunctional thin-walled shells.

11:40am **E2-1-ThM-12 Effects of Cathodic Currents on Mechanical and Corrosion Behaviors of Plasma Electrolytic Oxidation Coatings on 6061 Aluminum Alloy**, C. Tseng, Department of Materials Engineering, Ming Chi University of Technology, Center for Plasma and Thin Film Technologies, Ming Chi University of Technology, Taiwan; **Xianghe Wang**, Department of Materials Engineering, Ming Chi University of Technology, Taiwan

In this study, we fabricated the porous aluminum oxide (Al_2O_3) coatings on 6061 aluminum alloy by using plasma electrolytic oxidation (PEO) in alkaline sodium silicate solution under bipolar pulsed power mode with maxima anodic voltage at 500V, maxima cathodic voltage at 100V, duty cycle of 25%, pulse frequency in 1000 Hz, anodic current in 2A and cathodic current in 1~5A. The effects of cathodic current on microstructural, mechanical and corrosion behaviors of PEO coatings on 6061 aluminum alloy were investigated by X-ray diffractometer (XRD), scanning electron microscope (SEM), energy-dispersive X-ray spectroscopy (EDS), scratch test, pin-on-disk wear test and potentiodynamic polarization measurement. The XRD results show that the PEO coatings are mainly composed of $\alpha-Al_2O_3$ and $\gamma-Al_2O_3$. However, the ratios of $\alpha-Al_2O_3$ and $\gamma-Al_2O_3$ in PEO coatings are identical with various cathodic currents applied. The SEM images display that the thickness of PEO coating is increased with increasing cathodic current from 11mm (1A) to 17mm (5A) and surface porosity of PEO coating is decreased with increasing cathodic current from 16.2% (1A) to 9.4% (5A). The potentiodynamic polarization curves show that the PEO coatings, as compared to 6061 aluminum alloy, exhibit higher corrosion resistances in 3.5 wt% NaCl solution. However, the corrosion current density (I_{corr}) and passivation current density (I_{pass}) for PEO coatings are decreasing with increasing cathodic current applied. In summary, the thickness, porosity and corrosion resistance of PEO coatings on 6061 aluminum alloy can be significantly improved by controlling cathodic current applied.

12:00pm **E2-1-ThM-13 Metal/Oxide Nanolaminates by PVD-ALD: Maximum Strength-ductility for Al/ Al_2O_3** , **Thomas Edwards**, B. Putz, T. Xie, L. Vogl, H. Jansen, A. Groetsch, M. Watroba, J. Michler, Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland

The extent of the embrittlement in ductile-brittle multilayers often depends on the modulation period ($t_{brittle} + t_{ductile}$) as well as on the modulation ratio ($t_{brittle}/t_{ductile}$) [1]. In this work, ductile-brittle multilayers of Al/ Al_2O_3 /Al... were produced on Si substrates by a unique combination of atomic layer (ALD, Al_2O_3) and physical vapor deposition (PVD, Al) within a single deposition system. Using this ALD/PVD combination, neighbouring layer thicknesses can easily differ by one order of magnitude or more. In particular, the ability to deposit continuous sub-nm layers with ALD opens

up a wide range of otherwise unachievable modulation and thickness ratios. The thicknesses and structures of the ALD layers were verified by HR-TEM imaging of lift-outs. The amorphous oxide layer thickness is varied across the multilayer cross-section (0.1 nm – 10 nm) to study the effect on strength of the film as determined by microcompression, and on crack onset and propagation as a function of oxide layer thickness in tensile tested multilayer films. Similarly, the crystalline metal layer thickness is varied (10 nm – 150 nm) to optimise strength. The multilayer structure has good adhesion between individual layers and the oxide layers show increasing stretchability with decreasing film thickness. *In situ* TEM tensile loading was performed to evaluate the role of the amorphous-crystalline interfaces on dislocation motion in metallic layers, whilst microcompression at variable temperature and strain rate was used to quantify the activation parameters; this will be compared with molecular dynamics simulations currently underway. The thermal stability of such multilayer films was also studied up to $0.9 T_m$.

[1]K. Wu, J.Y. Zhang, J. Li, Y.Q. Wang, G. Liu, J. Sun, Acta Mater. 100 (2015) 344–358.

Tribology and Mechanical Behavior of Coatings and Engineered Surfaces

Room Town & Country B - Session E2-2-ThA

Mechanical Properties and Adhesion II

Moderators: Jazmin Duarte, MPI für Eisenforschung GMBH, Germany, Alice Lassnig, Austrian Academy of Sciences, Austria, Bo-Shiuan Li, National Sun-Yat Sen University, Taiwan

1:20pm **E2-2-ThA-1 Scratching the Surface: Understanding Plasticity Associated with Microscale Asperity Contacts**, Anna Kareer, University of Oxford, UK **INVITED**

When considering macroscale sliding, an understanding of the effect of plasticity is essential. Plastic behaviour directly affects the macroscopic wear processes and provides a quantitative mechanism of energy dissipation in friction. Recent nanotribology studies enable the investigation of sliding contacts at the scale of individual asperities, however, the instruments used to carry out these studies involve extremely low contact forces, which leave the surface unmodified by the sliding process. In this work we carry out nanoscratch experiments using a nanoindenter, in a single crystal copper sample. Nanoscratches are made with a constant normal force of 3 mN, which is sufficient to plastically deform the surface with a scratch penetration depth of approximately 250 nm. The residual deformation fields that surround the scratch are mapped using high resolution backscatter diffraction (HR-EBSD) and are compared to that of a statically loaded indent, in an attempt to understand the mechanisms of deformation. A physically based crystal plasticity finite element model (CPFEM) is used to simulate the lattice rotation fields and provides insight into the 3D rotation field surrounding a nanoscratch experiment as it transitions from a statically loaded indent to a kinetic scratch.

2:00pm **E2-2-ThA-3 Effect of Al/Ti ratio and Bias on Mechanical and Tribological Properties of AlTiN Coatings**, Jiri Nohava, Anton Paar TriTech SA, Switzerland; J. Sondor, LISS, a.s., Czechia

The effect of Al content and bias during deposition on the mechanical and tribological properties of the $(Al_xTi_{1-x})N$ coatings were characterized using nanoindentation, tribology and adhesion testing. The coatings were deposited on HSS steel substrate hardened to 64 HRC by cathodic ARC evaporation at 450°C on an industrial Platit PI 1011 equipment. This PVD machine allows simultaneous deposition from four targets: two Ti targets were used for deposition of TiN adhesion coating and two Al-Ti targets with three different target compositions (Al_{0.67}Ti_{0.33}, Al_{0.60}Ti_{0.40} and Al_{0.50}Ti_{0.50}) were used for deposition of monolayer top coating. The deposition parameters were the same for all samples except the DC bias which was set to -40V, -80V and -120V for each target combination.

The thickness of the coatings was 4.0 µm for 50/50 and 67/33 groups and 2.2 µm for the 60/40 group and the TiN adhesion layer was 0.6 µm. The wear tests, done with Ø6 mm alumina ball and contact pressure ~2200 MPa revealed similar friction coefficient of ~0.9 for all 60/40 samples whereas for the 50/50 and the 67/33 groups the friction coefficient was decreasing with decreasing bias: ~0.7 at -40V to ~0.5 at -120V. The wear rates decreased with decreasing bias: the lowest wear rates were obtained on the -120 V bias samples in the 50/50 and the 67/33 groups (up to three times increase in wear resistance at -120V compared to -40V in the 50/50 group). The effect of bias was less pronounced in the 60/40 group where only minor decrease of wear rate was observed. The wear performance seems to be related to the hardness of the sample in each group: hardness in the 50/50 and 67/33 groups increased from ~34 GPa and -40V bias to ~38 GPa for the -120V bias samples of the same group. Within the 60/40 group the hardness was approximately 31 GPa for all bias levels. The scratch test showed that samples with -40V bias from both 50/50 and 67/33 groups exhibited slightly lower critical load than the other bias levels from the same group and all 50/50 and 67/33 coatings showed more extensive scratch damage compared to the 60/40 coatings. This was evidenced by more extensive chipping on the edges of the scratches compared to scratches of the 60/40 group. Such scratch morphology is an indication of better cohesion of the 60/40 coatings.

The results show that the bias affects the hardness and adhesion only slightly but it strongly affects the wear rate: the lower the bias, the lower the wear rate. XRD measurements are currently underway to understand

the microstructure and mechanisms leading to better wear resistance of coatings deposited with the -120V bias.

2:20pm **E2-2-ThA-4 Effect of Nb and V Doped Elements on the Mechanical and Tribological Properties of CrYN Coatings**, İhsan Efeoğlu, G. Gülten, B. Yaylali, Y. Totik, Atatürk University, Turkey; P. Kelly, J. Malecka, Manchester Metropolitan University, U.K.

One of the most promising approaches to enhancing the tribological properties of engineering materials is to add transition elements to the structure. In this study, Nb and V doped CrYN thin films deposited by closed-field unbalanced magnetron sputtering (CFUBMS) system. The deposition parameters examined were target current (1, 1.5 and 2 A), deposition pressure (0.15, 0.25 and 0.35 Pa), pulsed frequency (100, 200 and 350 kHz) and duty time (0.43-5 µs). Taguchi L9 orthogonal design was used to arrange deposition process for each doped film. Microstructure, thickness, composition, hardness and tribological properties of Nb and V doped CrYN thin films were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), Energy dispersive spectroscopy (EDS), microhardness and pin-on-disc test, respectively. The bonding strength between the substrate and the film (adhesion) were analyzed by the scratch test. In Nb doped films, the maximum hardness value of 38± 0.5 GPa and the lowest friction coefficient of 0.36 were obtained. On the other hand, in V-doped films, the maximum hardness value was 29 ± 0.5 GPa, while the lowest friction coefficient of 0.11 were obtained. In addition, Nb doped films exhibited lower critical load values than V doped films, depending on the hardness and film thickness.

2:40pm **E2-2-ThA-5 Effect of Mo Interlayer on the Mechanical Properties and Tribology Behavior of Molybdenum Nitride Coatings Deposited by High Power Pulsed Magnetron Sputtering**, Yu-Che Fang, J. Huang, National Tsing Hua University, Taiwan

Transition metal nitride have been widely used as protective hard coatings owing to their superior mechanical properties, good corrosion resistance and high wear resistance. Molybdenum nitride (MoN_x) is one of the candidate materials for protective coating in tool industry due to the formation of self-lubrication Magnéli phases. In our previous study, the single phase γ-Mo₂N coatings were found to have good wear resistance, whereas adhesion issue occurred frequently during wear test. The addition of Mo interlayer may be a good remedy to enhance the adhesion of γ-Mo₂N coatings. Therefore, the purposes of this study were to investigate the effect of Mo interlayer on the mechanical properties and tribological behavior of γ-Mo₂N coatings deposited on AISI D2 steel substrate. The coatings were deposited using high power pulsed magnetron sputtering (HPPMS), where the 200-nm Mo interlayer was deposited by dc unbalanced magnetron sputtering (dc-UBMS). After deposition, the chemical compositions of the specimens were measured using an electron probe microanalyzer. The film thickness, cross-sectional microstructure and surface morphology of the specimens were examined by scanning electron microscopy. X-ray diffraction was used to characterize the structure and the texture of the coatings. The hardness and the elastic constant of the coatings were measured by nanoindentation. The residual stress of the coatings was measured by laser curvature method (LCM) and average X-ray strain (AXS) method. The surface roughness was measured by atomic force microscope (AFM). Scratch test and pin-on-disc test were used to determine the adhesion strength and wear resistance. The major concern of the adhesion strength will be evaluated by introducing different interlayer thickness, and the effect of interlayer on the residual stress and wear resistance will be discussed.

3:00pm **E2-2-ThA-6 Effect of Nitrogen Flow Rate on Tribological Behavior of TiN Thin Film on Ti-6Al-4V Deposited by Ion Plating Method**, K. Lan, An-Jia Chen, National Tsing Hua University, Taiwan

The purpose of this study is to investigate the tribological behavior of TiN thin films prepared by hollow cathode discharge-ion plating system on the titanium alloy Ti-6Al-4V. Although titanium alloys with excellent corrosion resistance and biocompatibility have been widely used in the field of biomedicine, such as Ti-6Al-4V, their hardness and wear resistance limited the durability in application. Various studies tried to improve the wear resistance of titanium alloys by using different techniques of surface treatment. However, delamination of surface coatings from titanium alloys can be observed easily which is correlated to unsatisfactory abrasion resistance and might be due to the existence of residual stress in the surface coating by PVD method. Recently, a good ionization rate ranging from 40 to 70% by ion-plating method can be attested which exhibited the potential to improve the abrasion resistance of deposited thin films and thus to overcome the problem of delamination. The objective of this study

is to improve the adhesion between TiN thin film and the substrate Ti-6Al-4V by using the hollow cathode discharge-ion plating (HCD-IP) method. The optimized nitrogen flow rate will be investigated. The structure and the texture of each sample are characterized by X-ray diffraction (XRD). The residual stress is assessed by laser curvature method (LCM) and average X-ray strain (AXS) method. The hardness of each sample is measured by nanoindentation. Scratch and Pin-on-disk tests will be carried out on TiN thin film samples to investigate adhesion and wear resistance, respectively.

3:20pm E2-2-ThA-7 Tailor the Tribological Behavior of TiN Coatings on D2 Steel by Adjusting Process Parameters during Deposition, *I-Sheng Ting, J. Huang*, National Tsing Hua University, Taiwan

The objective of this study was to tailor the tribological behavior of hard coatings by adjusting the process parameters during deposition. High residual stress has been considered as one of the crucial factors leading to the failure of hard coatings. From the perspective of energy, the stored elastic energy (G_s) in the coatings should be as low as possible. Once G_s reaches the fracture toughness of a coating (G_c), cracks may start to propagate and lead to the failure of coating. However, the energy-based theory ignores the stress distribution, especially the in-depth stress gradient in the coating, and cannot fully explain the coating failure. In fact, high compressive stress is conducive to impede the crack propagation. The relation between the stress distribution and the failure of coating has not been well understood. Since the generation of residual stress in hard coatings is closely related to the impingement of plasma species, the stress distribution can be tailored by adjusting the process parameters during deposition. In this study, TiN coating on D2 steel was selected as the model system to investigate the effect of stress distribution on the tribological behavior of hard coatings. TiN coatings were deposited on D2 steel using dc unbalanced magnetron sputtering, where the deposition parameters, working pressure and substrate bias were respectively adjusted during deposition to affect the impingement of plasma species. The stress of TiN coating was measured using the average X-ray strain (AXS) combined with nanoindentation methods [1-3], and the stress gradient was carried out using different X-ray incident grazing angles. The adhesion and wear resistance of TiN coating on D2 steel were evaluated using scratch test and pin-on-disk wear test, respectively. The stress distribution and the corresponding tribological behavior of TiN-coated D2 steel were found to depend on the impingement of plasma species. Tailoring the stress state of hard coatings shows great potential on the control of tribological behavior.

[1] C.-H. Ma, J.-H. Huang, H. Chen, *Thin Solid Films*, 418 (2002) 73-78.

[2] A.-N. Wang, C.-P. Chuang, G.-P. Yu, J.-H. Huang, *Surf. Coat. Technol.*, 262 (2015) 40-47.

[3] A.-N. Wang, J.-H. Huang, H.-W. Hsiao, G.-P. Yu, H. Chen, *Surf. Coat. Technol.*, 280 (2015) 43-49.

3:40pm E2-2-ThA-8 Micromechanics of Hydrogen Barrier Coatings During *In Situ* Hydrogen Charging, *Maria Jazmin Duarte Correa, H. Gopalan, J. Rao, P. Patil, C. Scheu, G. Dehm*, Max-Planck Institut für Eisenforschung, Germany

Hydrogen is a strong candidate to be the energy carrier of the future; however, its use also represents a challenge as it might cause material degradation through hydrogen embrittlement. Hydrogen barrier coatings represent, in this regard, an appealing option to prevent and/or slow down the hydrogen ingress into structural alloys that are susceptible to embrittlement. In this work we present our research on a 1-2 μm thick layer of Al_2O_3 and its effect as a hydrogen barrier coating on a Fe-Cr alloy. The mechanical stability of the coating was tested by nanoindentation and nanoscratching tests during hydrogen loading. For this purpose, we used our novel electrochemical cell design, the "back-side" charging approach, developed in-house to perform micromechanical testing during hydrogen charging [M. J. Duarte, et al., *J Mat Sci* 56 (2021) 8732-8744]. Hydrogen diffusion from the charged back-side towards the testing surface is quantified by permeation tests. Moreover, this unique method allows differentiating between the effects of trapped and mobile hydrogen, and performing well controlled measurements with different hydrogen levels monitored over time to consider hydrogen absorption, diffusion and release.

The mechanical behavior of the coating remained unaltered during the performed tests due to the slow hydrogen diffusion Al_2O_3 , which is about 9 orders of magnitude slower with respect to the Fe-Cr substrate, measured by the Kelvin probe technique. However, the accumulation of hydrogen at

the substrate-coating interface reduces the critical load required for the coating to crack, and might lead to local delamination during scratching. These mechanical analyses were complemented using atom probe tomography, confirming the presence of hydrogen close to the substrate/coating interface, and by transmission electron microscopy, used to reveal the microstructural changes related to hydrogen during scratching.

4:00pm E2-2-ThA-9 Nano-Scale Mechanical Characteristics of Epitaxial Stabilization ZrTiN/NbN Superlattice Coatings, *Pin-Yuan Lai, T. Ku, S. Hsu, P. Chen, J. Duh*, National Tsing Hua University, Taiwan

Protective hard coatings are widely used in industrial applications, especially under extreme conditions. In this regard, superlattices with epitaxial stabilization have shown to be a potential approach. Owing to the similar lattice parameter and crystal structure, the ZrTiN/NbN system was chosen. This superlattice system showed no cracks after indentation, possessing stronger crack resistance than both ZrTiN and NbN monolayers. The wear volume of multilayers was significantly lower than monolithic films. However, phase transformation of c-NbN to h-NbN occurred in relative thick sub-layer NbN. This phenomenon would deteriorate the wear resistance and elastic recovery of the coatings. HRTEM images confirmed that h-NbN led to the loss of epitaxial growth, and thus the coatings demonstrated polycrystalline structure. It was demonstrated that epitaxial ZrTiN/c-NbN superlattices can be controlled by adjusting the thickness ratio. The epitaxial structure exhibited not only favorable mechanical performance but also excellent tribological behavior.

4:20pm E2-2-ThA-10 Mechanical and Tribological Behavior of Nitrided AISI/SAE 4340 Steel Coated With NiP and AlCrN, *Ricardo Torres*, Pontifícia Universidade Católica do Paraná, Brazil; *M. Soares*, Universidade Tecnológica do Paraná, Brazil; *P. Soares*, Pontifícia Universidade Católica do Paraná, Brazil

Deterioration by wear and corrosion is a serious problem in the oil and gas industry. The presence of H_2S , CO_2 and chlorides with abrasive materials such as sand causes severe wear and corrosion. One strategy to mitigate steel surface deterioration is coating the steel surface with electroless NiP. The NiP coating on steel substrates requires an interdiffusion post-heat-treatment to create a metallurgical bonding between the steel substrate and the NiP deposit. However, this heat treatment causes a softening of the steel and the NiP deposit. This work investigated the following surface engineering strategies: i) NiP deposition in steel substrates that were previously nitrided followed by the NiP interdiffusion heat-treatment at 400°C or 610°C; ii) AlCrN PVD coating deposition on NiP layers on steel substrate that was previously nitrided followed by the NiP interdiffusion heat-treatment at 400°C or 610°C. Then, hardness and tribological behavior were determined. The tribological tests were performed in a ball-on-disk mode of tribometer applying a load of 20 N, a tangential speed of 25 cm/s. The counterpart used in the tribological tests was 6 mm diameter cemented carbide spheres; the total sliding distance was 1000 m. The friction coefficient was monitored throughout the tribological tests. The lowest wear rate was for the specimen with AlCrN PVD coating deposition on NiP layers on a previously nitrided steel substrate, followed by the NiP interdiffusion heat treatment at 610°C.

4:40pm E2-2-ThA-11 Designing Hydrogen-Free Diamond Like Multilayer Carbon Coatings for Superior Mechanical and Tribological Performance, *Muhammad Usman*, City University of Hong Kong

Diamond like carbon (DLC) coatings are in focus from a last few decades due to its exceptional mechanical and tribological properties. This class of coating borrows mechanical and tribological properties from diamond (sp^3) and graphite (sp^2) respectively. Therefore, high hardness, low coefficient of friction and wear rate are some of the intrinsic characteristics. Due to this unique combination, it finds wide applications in microelectromechanical systems (MEMS), automotive sector (tappet, camshaft, finger roller follower, camshaft sprocket, piston, piston rings), bearings, hip and knee joints, tools and dies, laser barcodes scanners, magnetic storage media. Researchers shifted from monolayer to multilayer architecture in DLC to achieve high hardness and resistance to plastic deformation (high toughness) simultaneously as these are inversely correlated [1-3]. Multilayers provide the optimum solution to this problem. Additionally, multilayers reduce compressive residual stress in the coating compared to hard monolayer [2]. The current research aims to evaluate the impact of bilayer numbers (bilayer thickness) on earlier mentioned properties. Therefore, new multilayer DLC coatings are designed with alternate hard and soft layers using closed field unbalanced magnetron sputtering (CFUBMS). Discrete sharp interfaces are produced by selecting two

Thursday Afternoon, May 25, 2023

different bias voltages. Hard to soft layer ratio is kept constant (1:1) for all specimens with fixed total coating thickness of $1\mu\text{m}$. 10, 20, 40 and 80 bilayers (shown in supplemental file) are deposited onto steel substrate having Cr/CrC_x interlayer. Raman spectroscopy, nanoindentation, nanoscratch and residual stresses are measured for all the specimens. Raman analysis depicts increasing I_D/I_G ratio and decreasing full width at half maximum (FWHM) trend by increase in bilayer numbers (decreasing bilayer thickness). Moreover, hardness and scratch resistance are directly proportion to number of bilayers. Residual stresses also increase with greater number of bilayers. This implies that graphitization is in direct relation to bilayer numbers and hardness increases potentially due to interlocking of graphitic clusters. 80 bilayers coating exhibited outstanding elastic and plastic deformation resistance. Hence, this design may offer the combination of high hardness and toughness in addition to wear resistance without introducing any other element or complex multilayer architecture, which require further investigations.

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 Mo₂N-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 Mo₂-N-Cu coatings that provides the best tribological response. Mo₂-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, Mo₂-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 CrAlN 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. Dehler, 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. *Ihsan Efeoglu, B. Yaylali, 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/hard-service 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 Magnetron Sputtering) 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.

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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, TiAl₂, Ti₄Si, and Ti₄Cu) 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 mm³/N·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 TiO₂ and Al₂O₃, 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 TiO₂ and SiO₂ 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 Effect of Sizing on the Adhesion Properties of Reclaimed Fiberglass Composites, *Nour Halawani*, Composite Recycling and LPAC - EPFL, Switzerland; *M. Anderson, P. Gallo, G. Perben*, Composite Recycling, Switzerland; *V. Michaud*, LPAC - EPFL, Switzerland

Recycling Glass Fiber Reinforced Plastics has been a challenge for decades. Currently, the most common solution for composite disposal is landfilling, which is the case for more than 90% of GFRP disposal worldwide. We have developed a thermal treatment based on pyrolysis which separates the fibers from the resin without the need for grinding or chopping the feedstock. The reclaimed fibers are then treated and integrated into new composites. To ensure a clean surface free from carbon impurities, the fibers are treated with a thermal cleaning method. After this process the resin, sizing, and the binder are removed from the surface and thus the surface still has impurities at the microscale which requires further treatment. The treatment of fibers requires additional cleaning, surface activation to increase the density of hydroxyl groups (-OH) on the fiber surface, and sizing application.

Sizing formulations are applied in a thin homogenous coating on fiberglass in the commercial production step. The main reason is to protect the fiber

surface during the production and post processing steps while creating different kind of fabrics. Additionally, the sizing is adapted to ensure good adhesion between the fibers and the matrix. In this work we aim to use the reclaimed fibers to produce new composites maximizing the mechanical properties in comparison with the initial composite, while keeping the treatment as minimal and benign as possible. We also aim to valorize the reclaimed fiberglass without losing their length.

For the sizing treatment, we apply two different kinds of sizing based on alkoxy silanes on fiber surfaces in order to study their effect on the adhesion as well as the final mechanical property of the obtained composite. A special combination of surface cleaning, surface activation and sizing application are applied and controlled using SEM and FTIR. The surface of the fibers are seen to be clean homogeneously. The flexural strength of the composite made up of untreated reclaimed fibers showed a decrease of 60 % whereas, when applying our surface treatment, we can see an improvement with a decrease of only 5 to 25% depending on the process and the sizing used. The adhesion of the treated fibers to the matrix is controlled using single fiber pull-off test validating our treatment procedure.

Bold page numbers indicate presenter

— A —

Ahmed, J.: E1-2-WeM-4, 5
 Alvarez, F.: E1-1-TuA-4, 3
 Amorim, F.: EP-ThP-11, 16
 Anderson, M.: EP-ThP-13, 16
 Ankamma, K.: E3-1-TuM-5, 1
 Ansell, T.: E1-2-WeM-2, 5
 Aouadi, S.: E3-1-TuM-9, 2; EP-ThP-1, 14
 Appleget, C.: E2-1-ThM-1, **8**
 — B —
 Barrie, J.: E2-1-ThM-1, 8
 Berkebile, S.: E1-2-WeM-4, 5; EP-ThP-1, 14
 Berman, D.: E3-1-TuM-9, 2; EP-ThP-1, 14
 Best, J.: E2-1-ThM-7, 9
 Bignoli, F.: E2-1-ThM-7, 9; EP-ThP-5, **14**
 Bobzin, K.: EP-ThP-2, 14
 Bondarev, A.: E3-1-TuM-4, **1**
 Brittain, R.: E3-1-TuM-8, 2
 Brognara, A.: E2-1-ThM-7, 9
 Broitman, E.: E3-1-TuM-8, 2; E3-2-WeA-9, **7**;
 EP-ThP-10, **15**
 Brühl, S.: E3-1-TuM-1, **1**
 Budnyk, S.: E1-2-WeM-12, 6
 Bull, S.: E2-1-ThM-5, **8**
 — C —

C, N.: E3-1-TuM-3, 1
 Cairns, E.: E3-1-TuM-9, **2**; EP-ThP-1, 14
 Caldwell, S.: EP-ThP-1, **14**
 Cammarata, A.: E1-1-TuA-4, 3
 Cavalcanti Pinto, H.: E1-1-TuA-9, **4**
 Cavaleiro, A.: E1-2-WeM-10, **5**
 Cemin, F.: E1-1-TuA-4, 3
 Chang, S.: E2-1-ThM-9, 9
 Chefer Apolinario, R.: E1-1-TuA-9, 4
 Chen, A.: E2-2-ThA-6, **11**
 Chen, P.: E2-2-ThA-9, 12
 Chen, Y.: E2-1-ThM-5, 8
 Choe, S.: EP-ThP-7, 15
 Choi, E.: EP-ThP-7, 15
 Chung, Y.: E1-2-WeM-4, **5**
 Cordill, M.: E2-1-ThM-3, **8**; EP-ThP-6, **15**; EP-
 ThP-8, **15**
 — D —

Daum, P.: E1-1-TuA-9, 4
 Dehm, G.: E2-1-ThM-7, 9; E2-2-ThA-8, 12
 Delfin, F.: E3-1-TuM-1, 1
 Desmarres, J.: E2-1-ThM-4, 8
 DEVOS, A.: E2-1-ThM-4, **8**
 Dixit, S.: E3-1-TuM-9, 2
 Djemia, P.: E2-1-ThM-7, 9; EP-ThP-5, 14
 Dockins, M.: EP-ThP-1, 14
 Duarte Correa, M.: E2-2-ThA-8, **12**
 Duh, J.: E2-2-ThA-9, 12
 Dunscombe, S.: E2-1-ThM-1, 8
 — E —

Echeverrigaray, F.: E1-1-TuA-4, 3
 Edwards, T.: E2-1-ThM-13, **10**
 Efeoğlu, İ.: E2-2-ThA-4, **11**; EP-ThP-3, **14**
 — F —
 Fang, Y.: E2-2-ThA-5, **11**
 Faurie, D.: E2-1-ThM-7, 9
 Fernández-Valdés, D.: EP-ThP-4, 14
 Figueroa, C.: E1-1-TuA-4, **3**
 Fleming, R.: E1-1-TuA-10, **4**
 Flores Cova, L.: E1-1-TuA-8, 3
 Flores Martínez, M.: E1-1-TuA-8, 3
 Folgner, K.: E2-1-ThM-1, 8
 Fukumasu, N.: E1-2-WeM-3, **5**
 — G —

Gachot, C.: E1-2-WeM-12, 6
 Gallo, P.: EP-ThP-13, 16
 Ganguly, R.: E3-1-TuM-3, 1
 Gay, P.: E1-2-WeM-13, **6**
 Ghidelli, M.: E2-1-ThM-7, 9; EP-ThP-5, 14

Gopalan, H.: E2-2-ThA-8, 12
 Greiner, C.: E1-1-TuA-9, 4
 Groetsch, A.: E2-1-ThM-13, 10
 Guerrero de León, A.: E1-1-TuA-8, 3
 Gülten, G.: E2-2-ThA-4, 11; EP-ThP-3, 14
 Gunda, R.: E3-1-TuM-5, **1**
 Gutnik, D.: EP-ThP-8, 15
 — H —
 Halawani, N.: EP-ThP-13, **16**
 Haviar, S.: E2-1-ThM-8, **9**
 Heim, D.: E3-1-TuM-1, 1
 Heo, S.: EP-ThP-7, 15
 Hodge, A.: E2-1-ThM-1, 8
 Hsu, S.: E2-2-ThA-9, 12
 Hsueh, C.: E1-1-TuA-5, 3
 Huang, J.: E2-2-ThA-5, 11; E2-2-ThA-7, 12
 — I —

Imrich, P.: EP-ThP-8, 15
 — J —
 Jansen, H.: E2-1-ThM-13, 10
 Jiao, V.: E2-1-ThM-1, 8
 Journot, T.: E1-2-WeM-13, 6
 Juang, J.: E2-1-ThM-10, **10**
 — K —

Kalscheuer, C.: EP-ThP-2, 14
 Kannan, R.: E3-1-TuM-3, 1
 Kareer, A.: E2-2-ThA-1, **11**
 Keckes, J.: E2-1-ThM-8, 9
 Kelly, P.: E2-2-ThA-4, 11; EP-ThP-3, 14
 KEMPE, P.: E3-2-WeA-10, **7**; EP-ThP-9, **15**
 Khan, A.: E1-2-WeM-4, 5
 Kim, J.: EP-ThP-7, 15
 Kim, W.: EP-ThP-7, 15
 Kleinbichler, A.: EP-ThP-8, 15
 Koch, O.: EP-ThP-2, 14
 Kozák, T.: E2-1-ThM-8, 9
 Kreiml, P.: E2-1-ThM-3, 8
 Ku, T.: E2-2-ThA-9, 12
 Kulkarni, A.: E1-2-WeM-2, **5**
 — L —

Lai, P.: E2-2-ThA-9, **12**
 Lan, K.: E2-2-ThA-6, 11
 Lassnig, A.: EP-ThP-8, **15**
 Lavalley, F.: E3-1-TuM-6, 2
 Lee, J.: E1-1-TuA-3, 3
 Leidens, L.: E1-1-TuA-4, 3
 Lenis Rodas, J.: E3-2-WeA-11, **7**; EP-ThP-12, **16**
 Li Bassi, A.: E2-1-ThM-7, 9; EP-ThP-5, 14
 Lim, J.: EP-ThP-7, 15
 Lin, M.: E2-1-ThM-6, 9
 Lin, Y.: E2-1-ThM-9, **9**
 Liu, S.: E1-2-WeM-4, 5
 Loehlé, S.: E1-2-WeM-5, **5**
 López-Liévano, A.: EP-ThP-4, 14
 Lou, B.: E1-1-TuA-3, 3
 — M —

Machado, I.: E1-2-WeM-3, 5
 Malecka, J.: E2-2-ThA-4, 11; EP-ThP-3, 14
 Mandal, S.: E3-1-TuM-3, 1
 Markovic Milosevic, I.: E1-2-WeM-13, 6
 Martin, T.: E1-2-WeM-4, 5
 Maurer, J.: E1-2-WeM-13, 6
 Meindlumer, M.: E2-1-ThM-8, 9
 Meneses-Amador, A.: EP-ThP-4, 14
 Meruvia, M.: EP-ThP-11, 16
 Michaud, V.: EP-ThP-13, 16
 Michels, A.: E1-1-TuA-4, 3
 Michler, J.: E2-1-ThM-13, 10
 Minea, T.: E1-1-TuA-4, 3
 Mitterer, C.: E2-1-ThM-3, 8
 Möbius, M.: EP-ThP-2, **14**
 Mochalin, V.: E1-1-TuA-1, **3**
 Morina, A.: E3-1-TuM-8, 2

— N —

Nascimento Pereira, J.: E1-1-TuA-9, 4
 Nguyen, T.: E2-1-ThM-6, **9**
 Nohava, J.: E2-2-ThA-3, **11**
 — O —

Oehler, M.: EP-ThP-2, 14
 Ouyang, F.: E2-1-ThM-9, 9
 — P —

Park, I.: EP-ThP-7, **15**
 Patil, P.: E2-2-ThA-8, 12
 Perben, G.: EP-ThP-13, 16
 Pereira, C.: EP-ThP-11, 16
 Pérez Alvarez, J.: E1-1-TuA-8, 3
 Perotti, B.: E1-1-TuA-4, 3
 Pichelbauer, K.: E1-2-WeM-12, 6
 Polcar, T.: E1-1-TuA-4, 3; E3-1-TuM-4, 1; E3-
 2-WeA-8, **7**
 Ponomarev, I.: E3-1-TuM-4, 1; E3-2-WeA-8, 7
 Putz, B.: E2-1-ThM-13, 10
 — R —

Rahmadtulloh, I.: E1-1-TuA-3, **3**
 Ramachandra Rao, M.: E3-1-TuM-3, **1**
 Ramanjaneyulu, K.: E3-1-TuM-5, 1
 Ramanujam, S.: E3-1-TuM-5, 1
 Ramchandra Reddy, A.: E3-1-TuM-5, 1
 Rank, M.: EP-ThP-2, 14
 Rao, J.: E2-2-ThA-8, 12
 Rao, S.: E3-1-TuM-3, 1
 Rashid, S.: EP-ThP-5, 14
 Rausch, M.: E2-1-ThM-3, 8
 Rego, R.: E1-2-WeM-3, 5
 Rivera Tello, C.: E1-1-TuA-8, **3**
 Rodriguez Ripoll, M.: E1-2-WeM-12, **6**
 Rodriguez-Castro, G.: EP-ThP-4, 14
 Romagnoli, D.: E3-1-TuM-6, **2**
 Rossi, E.: EP-ThP-5, 14
 — S —

Sales de Mello, S.: E1-1-TuA-4, 3
 Schachinger, T.: E1-2-WeM-12, 6
 Scheu, C.: E2-2-ThA-8, 12
 Sebastiani, M.: EP-ThP-5, 14
 Shergill, K.: E1-2-ThM-5, 8
 Sitzman, S.: E2-1-ThM-1, 8
 Soares, M.: E2-2-ThA-10, 12
 Soares, P.: E2-2-ThA-10, 12; EP-ThP-11, 16
 Sondor, J.: E2-2-ThA-3, 11
 Souza, G.: EP-ThP-11, 16
 Souza, R.: E1-2-WeM-3, 5
 Sudhakar Reddy, K.: E3-1-TuM-5, 1
 — T —

Tavares Avila, P.: E1-1-TuA-9, 4
 Ting, I.: E2-2-ThA-7, **12**
 Torres, H.: E1-2-WeM-12, 6
 Torres, R.: E2-2-ThA-10, **12**; EP-ThP-11, **16**
 Totik, Y.: E2-2-ThA-4, 11; EP-ThP-3, 14
 Trost, C.: EP-ThP-6, 15
 Tsai, C.: E2-1-ThM-9, 9
 Tschitschin, A.: E1-2-WeM-3, 5
 Tseng, C.: E2-1-ThM-12, 10
 — U —

Usman, M.: E2-2-ThA-11, **12**
 — V —
 Vidal-Torres, J.: EP-ThP-4, **14**
 Vital-Juarez, A.: E2-1-ThM-4, 8
 Vitu, T.: E3-2-WeA-8, 7
 Voevodin, A.: E3-1-TuM-9, 2; EP-ThP-1, 14
 Vogl, L.: E2-1-ThM-13, 10
 von Schleinitz, T.: E3-2-WeA-9, 7
 — W —
 Wang, C.: E1-1-TuA-3, 3
 Wang, H.: E2-1-ThM-6, 9
 Wang, J.: E2-1-ThM-9, 9
 Wang, Q.: E1-2-WeM-4, 5
 Wang, W.: E1-1-TuA-3, 3

Author Index

Wang, X.: E2-1-ThM-12, **10**
Watroba, M.: E2-1-ThM-13, 10
White, D.: E2-1-ThM-1, 8
Wu, G.: E3-1-TuM-8, **2**
Wu, H.: E2-1-ThM-6, 9

— X —
Xie, T.: E2-1-ThM-13, 10
— Y —
Yang, L.: E3-1-TuM-8, 2
Yaylali, B.: E2-2-ThA-4, 11; EP-ThP-3, 14
Yiu, P.: E1-1-TuA-5, 3

You, J.: E1-1-TuA-5, **3**
— Z —
Zak, S.: EP-ThP-6, 15
Zeman, P.: E2-1-ThM-8, 9
Zhang, T.: E2-1-ThM-6, 9
Zitek, M.: E2-1-ThM-8, 9