Tribology and Mechanical Behavior of Coatings and Engineered Surfaces
Room On Demand - Session E1
Friction, Wear, Lubrication Effects, and Modeling

E1-1 Multi-sensing Nano-wear with Electrical Contact Resistance and Friction Measurement*, **Ben Beake (ben@micromaterials.co.uk)**,* Micro Materials Ltd, UK*; T. Liskiewicz,* Manchester Metropolitan University, UK*; A. Harris,* Micro Materials Ltd.*; S. McMaster, A. Neville,* University of Leeds, UK

Wear begins at the asperities but typically the contact pressures acting on these are unknown in a standard macro-scale tribological test. In contrast, testing at the nano-/micro-scale ("single asperity tribology") enables the onset of wear to be studied conveniently and correlations with friction forces investigated to aid the design of surfaces with improved wear resistance.

Reciprocating contacts occur in a wide variety of practical wear situations including hip joints and electrical contacts. In optimising materials for improved durability in these contacts it is important that the contact conditions (e.g. sliding speed) can be reproduced. Hence, a capability for rapid high-cycle linear reciprocating nano-scale wear tests has been developed (NanoTriboTest) with automatic recording of friction loops, cumulative energy dissipation and electrical contact resistance. The design has high level of lateral rigidity providing the necessary stability to perform nano- or micro-scale wear tests for extended duration (e.g. several hours, up to 300 m sliding).

In this study, high cycle, up to 40 mm long track length reciprocating nano-wear tests have been performed on multilayer DLC coatings, and the biomedical alloys Ti6Al4V and 316L stainless steel. Stainless steel showed ductile response throughout the load range but an abrupt transition to higher friction and fracture-dominated wear after ~20 cycles occurred on Ti6Al4V. Friction and wear evolution in the test was compared to that in nano-fretting (gross slip) and nano-scratch (unidirectional) tests [1-3].

Improved detection of the onset of wear and the subsequent failure mechanisms was achieved by a multi-sensing approach where changes to electrical contact resistance were shown to correlate directly with the measured friction. Nano-wear tests of noble metal-noble metal contacts (Au-Au and Ag-Ag) showed much longer endurance than gold vs. steel contacts although occasional isolated failures were observed in 35000 cycle tests.

[1] Short note on improved integration of mechanical testing in predictive wear models, TW Liskiewicz et al, Surf Coat Technol 237 (2013) 212.

[2] Comparison of nano-fretting and nano-scratch tests on biomedical materials, BD Beake and TW Liskiewicz, Tribol Int 63 (2013)

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E1-2 Interlayer Design to Increase Adhesion of a-C Coatings onto C17200 Copper-Beryllium Alloy Surface*, **Marcos Dantas dos Santos (mdantas@usp.br)**, N. Fukumasu,* Polytechnic School of the University of Sao Paulo, Brazil*; A. Tschiptschin,* Metallurgical and Materials Engineering Department, University of Sao Paulo, Sao Paulo, Brazil*; R. de Souza, I. Machado,* Polytechnic School of the University of Sao Paulo, Brazil

Copper-beryllium (CuBe) alloys are widely used in the aeronautic and automotive industries due to its good thermal properties and corrosion resistance. However, reduced hardness and wear resistance can limit the use in manufacturing applications. To overcome these limitations, hard ceramic coatings can be applied to the tool surface, increasing the wear resistance, and reducing the friction coefficient. Among several coating options, amorphous carbon (a-C) coatings were selected in this work since this coating can present high hardness and a very low friction coefficient. Nevertheless, the main challenge in using a-C coatings onto CuBe alloys is the low adhesion between copper and carbon. This work focused on the development of a complex interlayer to increase adhesion under high contact, pure sliding, and dry tribological conditions. Two interlayer compositions (Ti/Si and Ti/TiN/Si) were analyzed based on the improved bonding between copper and titanium, while the amorphous silicon layer was applied to reduce the diffusion of carbon into the titanium-based layer. Pulsed Direct Current Magnetron Sputtering (pDCMS) system configuration was used to deposit the coatings using polycrystalline titanium, monocrystalline silicon, and polycrystalline graphite targets. The TiN interlayer was obtained by a reactive deposition process using argon and nitrogen gases. A polycrystalline graphite target was used to produce a hydrogen-free amorphous carbon coating over the tailored interlayers. The coatings were deposited onto C17200 CuBe discs, and silicon wafers substrates. Instrumented indentation tests were carried out in a Bruker Ti950 Triboindenter to evaluate coating hardness and elastic modulus. Increasing load scratch tests, using a diamond Rockwell C tip in a Bruker UMT-2 system, were conducted to evaluate coating adhesion to CuBe alloy substrate and measure friction coefficient and critical loads. Dry reciprocating ball-on-disk tribological tests were also conducted with this system, using two constant normal loads (10N and 20N) and a stroke of 4 mm. Scanning Electron Microscope (SEM), Raman spectroscopy and coherence correlation interferometry (CCI) were used to characterize the coatings and wear tracks after the tests. Results indicate that the Ti/TiN/Si compound interlayer improved the adhesion of the a-C coating, showing higher critical failure loads compared to other combinations. Also, ball on disc tests indicate the formation of a transfer layer at the ball surface, promoting reduced wear and failure of the coating when in contact with AISI5200 steel balls.

E1-3 Tribologically Enhanced Self-healing of Niobium Oxide Surfaces*, **Samir Aouadi (samir.aouadi@unt.edu)**, A. Shirani, J. Gu, B. Wei, D. Berman,* University of North Texas, USA

Activating a self-healing process is a viable approach for preventing the failure of ceramics experiencing mechanically-induced crack propagation. Previously, it was demonstrated that niobium oxide (Nb_2O_5) exhibits self-healing properties activated by the formation of Nb-Ag-O ternary oxide when heated above 945 °C in presence of silver. In this study, we explore the mechanism of lowering the high-temperature healing requirement by assisting the process of crack repair with a normal load and shear stresses. Specifically, we propose to use tribologically-induced local heating as a mechanism to enhance the self-healing ability of Nb_2O_5. During a pin-on-disk test, whereby a niobium oxide flat was sliding against a silver-coated ball, a sudden lowering of the coefficient of friction was observed at elevated temperatures (~600 °C). The better performance of the coating was associated with a surface reconstruction process initiated inside the wear track. Extensive characterization analysis of the wear track using energy-dispersive x-ray spectroscopy, Raman spectroscopy, and x-ray diffraction confirmed the presence of an Nb-Ag-O ternary oxide phase inside the wear track formed at elevated temperature. The formation of an Nb-Ag-O ternary oxide at a much lower than thermodynamically-required temperature suggests that the self-healing process can be initiated directly during mechanically induced stresses. Such a process is a new recipe for improving wear and crack resistance characteristics of ceramic components and maybe tuned to provide the desired frictional response.

E1-4 Computer Simulations of FCC Alloys Subjected to Dry Sliding as Basis for a Near-Surface Deformation Mechanism Map*, **Stefan Eder (stefan.eder@ac2t.at)**, M. Rodríguez Ripoll, U. Cihak-Bayr,* AC2T Research GmbH, Austria*; D. Dini,* Imperial College London, UK*; C. Gachot,* TU Wien, Austria

We study the microstructural response of five FCC CuNi alloys subjected to sliding with large-scale molecular dynamics simulations. The initial grains measure approximately 40 nm in diameter to ensure that plasticity is not dominated by grain boundary sliding, so our polycrystalline aggregate exhibits dislocation pile-up, twinning, and grain refinement analogous to polycrystals with much larger grains. We analyze the depth-resolved time development of the grain size, shear, twinning, and the stresses in the aggregate to produce a deformation mechanism map for CuNi alloys. This map captures the predominant microstructural phenomena occurring for a given composition and normal pressure, and will aid engineers in optimizing materials/surfaces to work within a required operating range. We compare tomographic visualizations of our atomistic model with focused ion beam images of the near-surface regions of real CuNi alloys that were subjected to similar loading conditions.

E1-5 From Surface to Sub-surface Contributions to Friction at the Nanoscale*, C. Menezes,* UFSC, Brazil*; V. Pavinato, L. Leidens,* UCS - Caxias do Sul University, Brazil*; F. Echeverrigaray, F. Alvarez,* UNICAMP, Brazil*; A. Michels, **Carlos Figueroa (carlos.cafiguer@gmail.com)**,* UCS - Caxias do Sul University, Brazil

The friction phenomenon is a complex manifestation of the nature originated in energy dissipation events owing to the lost work of non-conservative forces. In spite of different surface mechanisms describing the friction phenomenon at the nanoscale, the involved energy in such surface events is not enough to explain friction forces in wearless regime. Indeed, phononic, electronic and magnetic effects are not capable of providing a sufficient energy to explain friction forces. Thus, new contributions are mandatory to reach a satisfactory energy balance among friction mechanisms and lost work by non-conservative friction forces. The aim of the work is to establish sub-surface contributions to friction at the nanoscale. In this study, we report the friction forces at the nanoscale on iron nitride and oxide by nanoindentation followed of unidirectional sliding (NUS) and friction force microscopy (FFM). Two different experimental setups are reported. Moreover, the sub-surface elastic deformation due to indentation was modeled following the classical contact theory from Hertz by using the ABAQUS software. Firstly, the elastic deformation leads to reach elastic energies in the order of lost work of friction forces. Secondly, the sub-surface contributions seem to be more important than the surface contributions to friction at the nanoscale. We discuss these surface and sub-surface mechanisms by dissipation effects associated with surface phonon coupling and sub-surface energy-releasing due to elastic energy dissipation.

E1-6 Ni-based Self-Lubricating Laser Claddings for Hot Forming and High Temperature Vacuum Applications*, H. Torres,* AC2T Research GmbH, Austria*; B. Prakash,* Lulea University of Technology, Sweden*; **Manel Rodriguez Ripoll (Manel.Rodriguez.Ripoll@ac2t.at),*** AC2T Research GmbH, Austria

This work proposes a novel self-lubricating Ni-based laser cladding able to control friction at high temperatures while maintaining a superb wear resistance. The cladding microstructure consists of nickel dendrites surrounded by borides and homogeneously scattered pure silver pockets encapsulated within molybdenum and chromium sulfides that arise from the thermal decomposition of MoS_2 during deposition. This resulting microstructure is able to control friction from room temperature to 600 °C in ambient air and at least until 300 °C in vacuum. In ambient air, the friction reduction mechanism is determined by the silver and chromium sulfide pockets. Atomic force microscope investigations show that chromium sulfides have a high hardness and a low intrinsic friction. They additionally support further friction reduction by silver smearing due to their high hardness. At higher temperatures, the contribution of silver diminishes due to oxidation so that the contribution of chromium sulfides to self-lubrication is dominant.

The self-lubricating cladding shows decreased friction against Al-Si-coated 22MnB5 steel under hot stamping conditions. The presence of silver leads to a noticeable decrease in friction down to 0.3 during Al-Si coating break-up on the counter body, due to smearing next to the affected region. In the case of hot stamping against AA6082 aluminium alloy, the self-lubricating claddings in synergy with solid lubricants decrease friction and counter body wear at high temperatures compared to grade 1.2367 hot work tool steel commonly used in hot forming. These findings illustrate that the implementation of Ni-based self-lubricating laser claddings can lead to decreased costs while at the same time ensuring the quality of the hot stamped components.

In the case of high temperature vacuum performance, the presented self-lubricating cladding is able to effectively control and reduce friction down to a value of 0.25 against 440C martensitic stainless steel at room temperature and 300 °C by the smearing of silver over the chromium sulfides. This friction reduction mechanism is enhanced by the thermal softening of the pure silver phase at elevated temperatures, contrary to air atmosphere, where smearing is hampered by oxidation. This overall tribological performance makes the presented cladding also as potential candidate for space applications.

E1-7 INVITED TALK: The Thinnest of The Thin: Friction and Adhesion Behavior of Graphene and other Two-Dimensional Materials*, **Robert Carpick (carpick@seas.upenn.edu),*** University of Pennsylvania, USA **INVITED**

Two-dimensional materials provide a rich playground for exploring new and unexpected physical phenomena at the atomic limit of thickness, and provide opportunities for many applications including demanding tribological systems. This includes protective low friction coatings and additives, functional adhesive layers in flexible electronics, and nanoelectromechanical switches. I will focus on friction and adhesion behavior of nanoscale contacts with 2D materials measured with atomic force microscopy (AFM) and compared with molecular dynamics (MD) simulations. First, nanocontacts with 2-dimensional materials like graphene will be discussed. Friction is far lower than typical bare substrates, but depends on the number of 2D material layers present. An initial model attributing this to out-of-plane puckering [1] is now enhanced by MD showing a strong role of energy barriers due to interfacial pinning [2]. We also observe a large, order-of-magnitude increase in friction when graphene is fluorinated [3]. Using MD, we interpret this in the context of the Prandtl-Tomlinson (PT) model, where the potential energy surface (PES) corrugation controls friction. We also observe a non-monotonic dependence of friction on humidity for graphite. Using MD, this behavior is attributed to adsorbed water molecules that at low coverage act as pinning sites, but at high coverage form a quasi-ordered layer that provides a low friction incommensurate interface [4].

We also discuss 2D transition metal dichalcogenide (TMD) films including MoS_2. TMDs exhibit intrinsically low friction, although not as low as graphene. Like with fluorinated graphene, we attribute this to the (PES) corrugation [5]. To explore the temperature dependence, we use matched AFM and MD to study friction for tips sliding on monolayer and multilayer MoS_2 from cryogenic to elevated temperatures. Friction sometimes decreases dramatically with temperature (thermolubricity). However, the temperature dependence is at times weak, suggesting that atomic details of the contact can matter substantially, which we explore with MD simulations. Finally, new results from nanocontact experiments of 2D materials obtained *in situ* using transmission electron microscopy (TEM) will be presented. We observe nanoscale tip-on-tip contact and sliding behavior for few layer MoS_2, revealing intrinsic contact, adhesion, and friction properties of these ultrathin layers.

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E1-9 Tribological Properties of Vanadium-doped Coatings via Reactive Molecular Dynamic Simulations*, **Ilia Ponomarev (ponomili@fel.cvut.cz)**, T. Polcar, P. Nicolini,* Czech Technical University in Prague, Czech Republic

Friction and wear cause a quarter of losses of the global energy production. A well-known practical approach to reduce friction is to introduce another substance, called lubricant, to the contact surfaces. A variety of lubricants, both liquid and solid, are available on the market; the global lubricants market size was estimated at $128.51 billion in 2018.

However, friction reduction in some specific conditions may still be a challenge. Providing lubrication in oxidative environments at high temperatures, which is essential for certain technological applications, such as cutting tools, may serve an example of such a problem. Traditional lubricants, both liquid and solid, are unsuitable for the task due to their lack of oxidation resistance.

A possible solution of the problem – a hard and oxidation-resistant coating (e.g. TiN, Si_3N_4), containing a dopant that would, upon operation, diffuse to the surface of the coating and provide lubrication. Vanadium is a popular choice as such a dopant; in the operation conditions (above 700ºC, humid air) it is known to form oxides on the surface, which in turn melt, providing liquid lubrication. However, the exact mechanisms of oxidation and the effects of conditions on the resulting V_xO_y phases are not entirely clear.

We are studying the process of vanadium oxidation computationally. We use reactive molecular dynamics within Reactive Force Field (ReaxFF) approach. ReaxFF is an empirical potential, that is shown to be capable of performing at the Density Functional Theory (DFT) based methods level of accuracy, while consuming significantly less computational resources. ReaxFF enables nanosecond-long simulations for tens of thousands of atoms at the same computational cost, as hundreds of picoseconds for hundreds of atoms in DFT.

We develop a suitable ReaxFF parameterization and apply it to the oxidation simulations. We find vanadium pentoxide, V_2O_5, to be the predominant outcome of the oxidation. We study the effects of oxygen pressure, load, temperature and humidity on the oxidation rate and reaction outcome.

We further explore tribological properties of the V_xO_y phases yielded by oxidation and find out the mechanism of the vanadium oxide action. Sliding simulations in a wide range of conditions provide the answer to the question, how much V do we need for providing lubrication.

E1-11 Tribological Properties of Duplex PEO/Chameleon Coating on Aluminum Alloys*, Andrey Voevodin (andrey.voevodin@unt.edu)*, *J. Shittu, A. Shirani,* University of North Texas, USA*; A. Yerokhin,* University of Manchester*; A. Korenyi-Both,* Colorado School of Mines, USA*; J. Mogonye,* Army Research Laboratories, USA*; D. Berman, S. Aouadi,* University of North Texas, USA

In this study, plasma electrolytic oxidation (PEO) was used to create a porous oxide structure on AA 6082 aluminum alloys. This surface treatment resulted in the formation of a graded hard ceramic coating with a porous outer region. The porous regions were burnished with solid lubricants to create a hard/solid lubricant duplex multifunctional structure with an adaptive tribological response. The tribological proprieties of the duplex system was investigated by carrying out pin-on-disk and reciprocating wear tests in humid air using a range of temperatures, loads, and sliding speeds. A low friction coefficient was maintained for all test conditions, suggesting the self-adaptive nature of the selected solid lubricant mixture. High temperature pin-on-disk tests were carried at 400 ° C with an apparatus equipped with an *in situ*Raman system to monitor real time chemistry changes in the wear track. *In situ*Raman spectroscopy provided new insights into the tribochemical processes that occur at elevated temperatures for different loads and sliding speeds.

E1-13 Analysis of Coating Layers and Defects Using Atomic Force Microscopy*, Stefan Kaemmer (stefan@parksystems.com)*, *G. Mendoza,* Park Systems Corporation, USA

Coatings provide important roles in industrial environments. They can protect the underlying material from harsh environments or improve the tribological properties of machine parts as an example [1]. Even small defects or imperfections can act as a failure center. The inspection of the coating quality becomes therefore an important step during the development and production of the coating process.

Atomic Force Microscopy (AFM) is a well-established technique for analysis of surface morphology with sub-nanometer resolution. It has become a routine tool in material research and semiconductor manufacturing for quality control of surfaces. For standard morphological analysis, the AFM is generally operated in Non-Contact mode, which allows for non-destructive, quantitative, three-dimensional analysis of the surface topography (figure 1). However, AFM does not only allow for the morphological analysis of surfaces. By combining AFM with other techniques information like the frictional properties, electrical conductivity, surface charges, magnetic properties etc. can be extracted and mapped with nanometer resolution as well. A recent development leverages the AFM resolution capabilities and enables the determination of the nanomechanical properties, like modulus and adhesion. This so called 'PinPoint" mode is based on fast force-distance curves that are executed at each pixel [3]. Figure 2 depicts a basic force curves and some of the information that can be extracted.

We will discuss applications and highlight how PinPoint AFM can help to determine the difference between a scratch before and after coating.

References

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