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

Mechanical Properties and Adhesion II

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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 TriTec SA, Switzerland*; J. Sondor,* LISS, a.s., Czechia

The effect of Al content and bias during deposition on to 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 Pl 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 (Al0.67Ti0.33, Al0.60Ti0.40 and Al0.50Ti0.50) 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. Yaylalı, 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 isto 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-Mo_2N 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-Mo_2N coatings. Therefore, the purposes of this study were to investigate the effect of Mo interlayer on the mechanical properties and tribological behavior of γ-Mo_2N 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-desk 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 Tribological Behavior of TiN Thin Film Deposited by Magnetron Sputtering System on Ti6Al4V with different α/β Grain Sizes*,*** *K. Lan,* ***An-Jia Chen,*** National Tsing Hua University, Taiwan

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. According to literature, most failure of thin film on Ti6Al4V might be related to a low hardness and a low elastic modulus of Ti6Al4V compare to the properties of the hard coating deposited on the substrate. Moreover, the hardness of Ti-6Al-4V seems to varied with its grain size of α and β phase. Thus, the objective of this study is to investigate the tribological behavior of TiN thin films prepared by magnetron sputtering system on the titanium alloys Ti-6Al-4V with varied grain sizes of α and β phase. The α/β grain sizes of Ti-6Al-4V 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 composes of the TiN and Ti-6Al-4V substrate is measured by nanoindentation and Vickers hardness. Scratch and Pin-on-disk tests will be carried out on TiN thin film samples to investigate adhesion and wear resistance, respectively.

Keywords: nitride, titanium alloy, tribology, adhesion

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.

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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 occured 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**,* Pontificia Universidade Católica do Paraná, Brazil*; M. Soares,* Universidade Tecnologica do Parana, 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 H2S, CO2 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 400oC or 610oC; ii) AlCrN PVD coating deposition on NiP layers on steel substrate that was previously nitrided followed by the NiP interdiffusion heat-treatment at 400oC or 610oC. 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 610oC.

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 different bias voltages. Hard to soft layer ratio is kept constant (1:1) for all specimens with fixed total coating thickness of 1µm. 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.

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