Tribology and Mechanical Behavior of Coatings and Engineered Surfaces
Room On Demand - Session EP

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

EP-1 About the Impossibility of a Mathematical Relationship between Hardness Values Measured by Vickers and Instrumented Nanoindentation Techniques, ***Esteban Broitman (esteban.daniel.broitman@skf.com)***, SKF Research & Technology Development Center, Netherlands

The hardness of a solid material can be defined as a measure of its resistance to a permanent shape change when a constant compressive force is applied. At macro- and microscale, the Vickers hardness test is assessed from the size of an impression left under a load by a four-sided pyramid-shaped diamond indenter. The Vickers hardness number, HV, is then calculated as the indenter load L divided by the actual surface area of the impression A_c measured after the indentation. On the other hand, the instrumented nanoindentation hardness H_{IT}, using a three-sided pyramid-shaped diamond indenter, is calculated as the maximum indenter applied load L_{max} divided by the projected area of contact at maximum load A_{pml}, i.e., during the indentation [1].

There are many publications where authors try to compare their coating hardness values measured by nanoindentation with bulk hardness values that have been measured by Vickers tests. The comparison is usually made through a formula that is supposed to give an exact mathematical equivalence of hardness values between both methods: HV = 0.09 H_{IT}, with HV having units of kgf/mm^2 and H_{IT} having units of MPa. In this work, I demonstrate that this exact equivalence can be established only for hardness values of materials with 0% indentation elastic recovery. In other cases, I will show that it is impossible to establish such mathematical relationship.

[1] E. Broitman, "Indentation Hardness Measurements at Macro-, Micro-, and Nanoscale: A Critical Overview," *Tribology Letters,* vol. 65, p. 23, 2017. (Open Access Article)

EP-2 Substrate Influence on the Adhesion of Metallic Films, ***Megan J. Cordill (megan.cordill@oeaw.ac.at)***, Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Austria; *P. Kreiml,* Erich Schmid Institute for Material Science, Austrian Academy of Sciences, Austria

Flexible and foldable electronics are becoming more visible for consumers. These devices are often manufactured with metallic films or islands deposited onto compliant polymer substrates. More research is needed to fully understand how to control the metal-polymer interface of the vital interconnecting metal lines found in flexible electronics to improve reliability. While the methods to quantify the interface adhesion are available, a direct comparison of the adhesion of a standard metallic films on different substrates has not been performed. Our results demonstrate that a sputtered Ti adhesion interlayer does not improve the adhesion of Ag films to polyimide (PI) or polyethylene naphthalene (PEN). In addition, the Ag-PI interface had a higher adhesion energy relative to the Ag-PEN interface due to the different polymer substrate chemistries which influence the interface structure and chemistry.

EP-3 Effects of Sputtering Gas Systems on the Preparation of a-BN Films using RF Sputtering, ***Yuki Yamada (s16A3132FM@s.chibakoudai.jp)***, *T. Markuko,* Chiba Institute of Technology Graduate School, Japan; *M. Imamiya,* Hana Saidan, Japan; *Y. Sakamoto,* Chiba Institute of Technology, Japan

Mechanical properties and the structure of boron nitride (BN) are similar to carbon materials. In particular, chemical inertness of BN such as excellent oxidation resistance and reaction with iron-based materials is better than carbon. In addition, structure of a-BN (amorphous BN) is similar to amorphous carbon such as Diamond-like carbon. a-BN is considered to have excellent tribological properties. However, little has been reported on tribological property of a-BN films. So, in this research, preparation of a-BN films by sputtering method and evaluation of the tribological property were investigated.

BN films were prepared by RF sputtering using Ar, Ar-N_2, and Ar-N_2-H_2 as sputtering gas systems. h-BN was used as a target. RF power and pressure were 500W and 0.5Pa, respectively. Deposits were evaluated using Raman spectroscopy. The tribology properties were investigated using a ball on disk friction test.

The peaks of sp^2-BN (near 1370 cm^{-1}) and a-BN (near 1600 cm^{-1}) were recognized in the Raman spectra prepared under all conditions. In addition, peak of sp3-BN (near 1310 cm^{-1}) was recognized in the Raman spectrum of Ar-N_2-H_2.

As a result of the friction test, it was confirmed that a low friction coefficient of 0.1 was exhibited at sputter gas Ar-N_2-H_2. In contrast, high friction coefficient was exhibited at sputter gas Ar and Ar-N_2. From the wear depth of the sample after the friction test, it was confirmed that the wear depth was the deepest in Ar and the shallowest in Ar-N_2. The difference in the wear depth is considered to be caused by the difference in the wear mechanism.

As a result of Raman spectroscopy of the adhesion to the ball after friction test, the peak of H_3BO_3 was recognized under conditions exhibiting high friction coefficient (sputtering gas; Ar, Ar-N_2). Conversely, no peak of H_3BO_3 was recognized under the condition exhibiting low friction coefficient (sputtering gas; Ar-N_2-H_2). Therefore, the high friction coefficient exhibited in Ar and Ar-N_2 are considered to be due to the formation of H_3BO_3 during the friction test. Furthermore, low friction coefficient exhibiting in Ar-N_2-H_2 is caused by no formation of H_3BO_3 during the friction test.

In conclusion, tribology properties of a-BN were varied on the structure by using of different sputtering gas systems, and low friction coefficient was exhibited at Ar-N_2-H_2 sputter gas.

EP-4 Tribological Properties of Sputter-deposited Mo Films on Polyimide, ***Edyta Kobierska (edyta.kobierska@unileoben.ac.at)***, *S. Hirn,* Montanuniversität Leoben, Austria; *M. Cordill,* Erich Schmid Institute for Material Science, Austrian Academy of Sciences, Austria; *R. Franz, M. Rebelo de Figueiredo,* Montanuniversität Leoben, Austria

In the last decade, the shift from rigid to flexible electronics has gained momentum and is mainly driven by display and touch panel technologies that are developed for flexible substrates like polymers or textiles. Unlike rigid electronics, thin film materials used in flexible electronics must withstand various static and dynamical loading conditions in order to ensure that the flexible display remains operational for a sufficiently long period of time. Tribological loading conditions are among them, in particular in the case of wearable electronics, but have only been scarcely studied in literature. Therefore, the tribological propertied of Mo films which were deposited on polyimide substrates with a thickness of 125 µm were analyzed. The Mo films were synthesized by high power impulse magnetron sputtering to a thickness of about 1 µm. To induce different residual stress states in the Mo films, two deposition distances (8 and 14 cm) and two Ar pressures (0.5 and 1 Pa) were used. The tribological tests were performed in ball-on-disk configuration with 1000 laps applying a load of 0.244 N. As counterparts, different materials were chosen including Al_2O_3, 100Cr6, PEEK (polyether ether ketone), POM (Polyoxymethylene) and NBR50 (nitrile butadiene rubber) to test the Mo films in different tribological contact situations. The recorded coefficient of friction (COF) was highest in the tests against NBR50 with values up to 3 in the beginning of the test and a subsequent steady decrease. The COF in the tests against the other counterpart materials was generally between 0.5 and 1.5. In terms of wear, higher wear was observed for in the tribological tests against counterparts of high hardness, i.e. Al_2O_3 and 100Cr6. As expected, abrasive wear mechanisms are active in these cases as observed from images of the wear tracks recorded with a 3D laser confocal scanning microscope and a scanning electron microscope. Predominant adhesive wear was noticed in the tests against the polymers and the rubber counterpart. The obtained results generally serve as a basis to explore the tribological behavior of thin films on flexible substrates like polymers.

EP-5 Tribocorrosion Behavior of Boride Coating on Cocrmo Alloy Produced by Thermochemical Process in 0.35% NaCl Solution, *A. Rentería,* Universidad de Guadalajara, México; ***Marco Antonio Doñu-Ruiz (marckdr_69@hotmail.com)***, Universidad Politecnica del Valle de Mexico, México; *M. Flores-Martinez,* Universidad de Guadalajara, México; *S. Muhl,* Universidad Nacional Autónoma de México, México; *N. Lopez-Perrusquia,* Universidad Politecnica del Valle de México, México; *E. García,* CONACYT - Universidad de Guadalajara, México

This work presents the corrosion and tribocorrosion studies of the CoB and Co_2B layer on CoCrMo alloy surfaces, produces by the thermochemical process. The boriding process was carried out at 850°C per 2 hrs, using dehydrated boron past such as boron source. The boride layers were characterized with XRD, SEM and optical profilometry in order to determine the structure, surfaces morphology and roughness, respectively.

A solution of NaCl at 3.5% was used to study the corrosion and tribocorrosion performance of the coated and uncoated surfaces. The tribocorrosion tests were carried out in a sliding-contact system with reciprocal movement, using a ball of Al_2O_3 of 10 mm such as counterbody. The corrosion test showed that the boride surfaces presented a higher tendency to the corrosion with higher E_{corr} and I_{corr} than the uncoated surfaces, nevertheless, in the tribocorrosion characterization this surface had similar kinetic friction coefficient and lower wear volume than the uncoated surfaces.

EP-6 Composite Coating on Cu Prepared by Plasma Electrolytic Aluminating, *C. Zhao, J. Sun, R. Cai,* ***Xueyuan Nie (xnie@uwindsor.ca),*** University of Windsor, Canada*; J. Tjong,* Ford Motor Company, Canada*; D. Matthews,* University of Twente, Netherlands

Metal-ceramic composite coatings were successfully prepared on pure copper by the plasma electrolytic aluminating (PEA) process. The mechanism of PEA processing and the microstructure of the prepared coatings were studied by scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), X-ray photoelectron spectroscopy (XPS) and X-ray diffraction (XRD) analyses. It has been revealed that the $Al(OH)_3$ passive film formed on the copper surface was indispensable for plasma discharges. XRD analysis indicated that the prepared coating consists of metallic Cu, Cu_2O, and Al_2O_3. Tribological tests reveal that the PEA treatment significantly increased the wear resistance in dry-sliding conditions, which could be attributed to the high hardness of the prepared coating. The wear mechanism changed from the adhesive wear of the pure copper to abrasive wear of the steel ball after the PEA treatment. Electrochemical tests show that the coated copper has much better performance against corrosion in 3.5 wt.% NaCl solution at room temperature.

Keywords:plasma electrolytic aluminating; copper; wear; electrochemical corrosion

EP-7 A Numerical-Experimental Study of Borided AISI 316L Steel Under Cyclic Contact Loading, ***Daybelis FERNÁNDEZ (ingday1989@hotmail.com),*** *O. DE LA ROSA, G. Rodríguez-Castro, A. Meneses-Amador,* National Polytechnic Institute, Mexico*; A. LÓPEZ-LIÉVANO, A. Ocampo-Ramírez,* Instituto Sanmiguelense, Mexico

Borided AISI 316L steels under cyclic contact loading were evaluated. Boriding was carried out by two powder-pack processes: continuous and interrupted process. surface hardened by the boriding process. Boriding processes were developed at 1173 K for 1 h (continuous process) and 4 h (interrupted process). A Fe_2B monophase layer was obtained by the interrupted boriding, while a FeB/Fe_2B bi-phase layer was formed by the continuous boriding. Cyclic contact tests were performed on a servo-hydraulic testing machine by cyclic loading of a sphere on the borided steel surface. Circumferential cracks because of the applied critical load (monotonic load) were observed at the borided steel surface. Subcritical loads with a frequency of 6 Hz were applied on the borided steel surface to evaluate the evolution of the damage caused. Stress field generated at the borided steel surface because of cyclic spherical contact was obtained by the finite element method. Interrupted boriding process showed a better resistance to cyclic contact loading than the continuous boriding process.

EP-9 Novel Micromechanical Approaches to Understand the Influence of Hydrogen on Materials Behavior, ***Jazmin Duarte Correa (j.duarte@mpie.de),*** *J. Rao,* Max-Planck-Institut für Eisenforschung GmbH, Germany*; X. Fang,* Technische Universität Darmstadt, Germany*; G. Dehm,* Max-Planck-Institut für Eisenforschung, Düsseldorf, Germany

The understanding of hydrogen interactions with different features (e.g. dislocations, grain boundaries, precipitates, etc.) in alloys and composites is essential either to control and benefit from the hydrogen technology, or to prevent the destructive outcome of hydrogen embrittlement. Failure mechanisms initiate at the atomic scale with hydrogen absorption and further interaction with trap binding sites or defects. Nanoindentation and related techniques are valuable tools to study independently such mechanisms due to the small volume probed. Even more, in situ testing while charging the sample with hydrogen can prevent the formation of concentration gradients due to hydrogen desorption.

Two custom electrochemical cells were built for in situ hydrogen charging during nanoindentation of the sample (Figure 1): “front-side” charging with the sample and indenter tip immersed into the electrolyte, and “back-side” charging where the analyzed region is never in contact with the solution and therefore the observed effects are only due to hydrogen. We discuss the advantages and disadvantages of both approaches during the study of the hydrogen effect on the mechanical behavior and incipient plasticity in bcc FeCr alloys. The newly developed back-side charging technique allows overcoming surface degradation that might occur during front-side charging. The presence of hydrogen on the top analyzed surface (Figure 1b) was assessed by Kelvin probe measurements, showing a fast hydrogen diffusion rate towards the upper surface as well as a pronounced release flow for the analyzed Fe-Cr alloys. This approach is being extended to the study of coatings, with especial interest at interfaces, often becoming hard trapping sites for hydrogen. These studies are therefore complemented with powerful characterization techniques (microscopy and analytics) to understand the role of hydrogen on the materials failure.

EP-10 Enhancement in Dry Cutting Performance and Tribological Characteristics of Amorphous Carbon and Bimetal Nitride Coatings Deposited by HiPIMS Technology With Positive Pulses, ***David Matthews (d.t.a.matthews@utwente.nl)***, University of Twente, Netherlands*; R. Ganesan,* University of Sydney, Australia*; I. Fernandez-Martinez,* Nano4Energy, Spain*; M. Stueber, S. Ulrich,* Karlsruhe Institute of Technology (KIT), Germany*; D. Mckenzie, M. Bilek,* University of Sydney, Australia

High power impulse magnetron sputtering (HiPIMS) technology with positive pulses was employed in the production of a-C, AlCrN and AlTiN coated tungsten carbide inserts for end milling applications. The results reveal positive effects for increasing productivity, improved surface finish and thickness uniformity, as well as enhanceddry cutting performance. Although HiPIMS is a proven PVD technology to deposit dense coatings it is well-known that drawbacks in coating production by HiPIMS include lower deposition rates, higher residual stress and lower adhesion when compared to techniques such as Cathodic-arc and pulsed-cathodic arc. In this work, the advantage of employing positive pulses to modulate ion fluence towards the coating substrate to modify the film properties is presented. Experimental results showed that a minimum threshold pulse duration of HiPIMS voltage and magnitude of HiPIMS current is required to exploit the full advantage of positive pulses. The optimized parameters for positive pulses yield increased coating deposition rates, reduced argon content and improved adhesion properties. The wear resistance of the films has also been substantially improved. Comparative studies on the positive pulse tailored coatings sliding in air and vacuum environments at different speeds elucidated the advantage of applying optimized positive pulse parameters on the wear behaviour of the coatings.

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