### Tribology and Mechanics of Coatings and Surfaces Room Golden State Ballroom - Session MC-ThP

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

MC-ThP-1 Influence of Cobalt Content on the Adhesion of TiAIN and AITIN/TISIN Coatings on WC-Co Substrates, Bruna Michelle de Freitas (bruna.michelledefreitas@gmail.com), R. Diego Torres, D. Stolle da Luz Weiss, P. Cesar Soares Junior, C. Augusto Henning Laurindo, Pontificia Universidade Católica do Paraná, Brazil; F. Lacerda Amorim, Pontificia Universidade Catolica do Paraná, Brazil

Coatings based on Ti-Al-N-Si, deposited by the physical vapor deposition (PVD) process, aim to increase the working life of machining tools. In this way, the mechanical properties of the substrate interfere with the adhesion of these coatings, reflecting on their performance. Cemented carbide, one of the most used materials for cutting tool manufacturing, is a composite consisting of tungsten carbide (WC) plus a binder phase. The binder content, typically cobalt (Co), defines its main characteristics: hardness, elastic modulus and determining its application [1]. However, the effect of cobalt composition in the cemented carbide substrate and how its variation affects the mechanical properties and adhesion of PVD coatings is not extensively investigated [2]. Therefore, the objective of this study is to evaluate the adhesion and mechanical properties (hardness (H), elastic modulus (E), and the H/E ratio) of Ti-Al-N-Si-based coatings (TiAlN and AlTiN/TiSiN) deposited by the PVD process on cemented carbide substrates with different Co concentrations (6, 8, and 10%). The surface properties of the substrates and coatings were assessed using scanning electron microscopy (SEM) with an attached energy-dispersive X-ray spectroscopy (EDS) system, X-ray diffraction (XRD), and roughness measurements. For the evaluation of mechanical properties, nanoindentation tests were performed, and adhesion was evaluated through indentation testing and scratch testing. The results show that the hardness and elastic modulus of the substrates are affected by the Co content, and the AlTiN/TiSiN coating has the highest hardness due to the presence of Si in its composition, along with higher roughness from the deposition process. In general, higher Co content in the substrate negatively affects adhesion. Through the scratch test, it was observed that the TiAIN coating has better adhesion to the substrate. Additionally, for a higher H/E ratio, there is greater adhesion for both coatings (TiAIN and AlTiN/TiSiN), and this adhesion is higher in cemented carbide substrates with low Co content.

[1] CHEN C. et al., Additive manufacturing of WC-Co cemented carbides: process, microstructure, and mechanical properties, Additive Manufacturing, 2023.

[2] CHEN Y. et al., Cohesive failure and film adhesion of PVD coating: Cemented carbide substrate phase effect and its micro-mechanism, International Journal of Refractory Metals and Hard Materials, 2023.

#### MC-ThP-3 Application of *In Situ* Hydrogen Charging During Micromechanical Testing of Thin Films, *Szilvia Kalacska (szilvia.kalacska@cnrs.fr)*, CNRS LGF, Mines St. Etienne, France

Understanding mechanisms of deformation in thin films at the sub-micron scale is the key for designing new compositions for industrial applications. It requires the determination of strains/stresses [1], dislocation distribution [2] and the overall microstructure evolution, which is often extremely challenging. Microstructural processes during external mechanical loading are hard to observe due to the complex multiscale nature of the phenomenon. If hydrogen is present in the solid, it can cause embrittlement or enhanced cracking, when the material is subjected to stress. This would eventually lead to the reduced lifetime or critical failure of the component. Although it is known for a long time that hydrogen causes degradation of mechanical performance in metals, the microscale mechanisms remain a subject of debate. Direct H-detection within the lattice is an extremely challenging task, (continuous diffusion and outgassing issues). Microstructure observations are still mostly performed post mortem on bulk samples.

In situ H-charging is therefore essential for thin film experiments. Samples can be loaded electrochemically through the back surface [3], using a cell compatible with high-vacuum (HV) scanning electron microscopes (SEM). This way, H diffuses into the lattice from the back, avoiding contamination to the surface of interest. The developed system will be presented, focusing

on the coupling of the cell with the nanodeformation stage by performing nanoindentation experiments on H-charged thin films.

### References:

[1] S. Wang, S. Kalácska, X. Maeder, J. Michler, F. Giuliani, T. B. Britton, The effect of  $\delta$ -hydride on the micromechanical deformation of a Zr alloy studied by in situ high angular resolution electron backscatter diffraction, *Scripta Materialia* 173 (2019) 101-105. doi: 10.1016/j.scriptamat.2019.08.006

[2] S. Kalácska, *et al.*, Investigation of geometrically necessary dislocation structures in compressed Cu micropillars by 3-dimensional HR-EBSD, *Materials Science and Engineering A* 770 (2020) 138499. doi: 10.1016/j.msea.2019.138499

[3] J. Kim, C. C. Tasan, Microstructural and micro-mechanical characterization during hydrogen charging: An in situ scanning electron microscopy study, *International Journal of Hydrogen Energy* 44 (12) (2019) 6333-6343. doi: 10.1016/j.ijhydene.2018.10.128

#### MC-ThP-4 Shrouding Gas Plasma Deposition Technique for Generating Wear Resistant ZnO/WS<sub>2</sub> Composite Films on PEEK, Dietmar Kopp (dietmar.kopp@joanneum.at), Leobner Straße 94a, Austria

In this study, zinc oxide/tungsten disulfide (ZnO/WS<sub>2</sub>) composite films were generated by an atmospheric pressure plasma jet (APPJ) equipped with a shrouding gas attachment on polyether ether ketone (PEEK) discs. The friction and wear properties of the ZnO/WS<sub>2</sub> composites sliding against 100Cr6 counterpart balls were intensively investigated by using a rotational ball-on-disk setup under dry sliding conditions at ambient room conditions. The deposited and worn coating areas were observed with a scanning electron microscope (SEM). The results indicated that low friction ZnO/WS<sub>2</sub> composite films have the potential to protect PEEK against mechanical motion. However, the tribological performance of the coatings are strongly dependent on the plasma-process settings (i.e. plasma current, dwell time of the powder particles in the plasma jet). In fact, there is a significant tribological improvement of the composite films in contrast to the uncoated PEEK by a factor of three.

MC-ThP-5 Wear and Corrosion Characterization and Parametric Optimization of Nb-doped Hydrogenated Diamond-like Carbon (a-C:H) Coatings, Ihsan Efeoglu (iefeoglu@atauni.edu.tr), Y. Totik, G. Gulten, B. Yaylali, M. Yesilyurt, Atatürk University, Turkey; R. Gunay, G. Kara, B. Altintas, TUSAS ENGINE INDUSTRIES (TEI), Turkey

This study focuses on enhancing the wear and corrosion resistance of AISI 4130, a chromium-molybdenum alloy steel, through the application of a functional coating. Targeting various industrial uses, notably in the aerospace and automotive industries, the research aims to improve the durability and performance of AISI 4130. As the functional coating, niobium-doped hydrogenated diamond-like carbon (a-C:H:Nb) coatings were deposited using a closed-field unbalanced magnetron sputtering technique under various parameters, which were systematically optimized following the Taguchi L<sub>9</sub> orthogonal array method. The microstructural properties of the coatings were analyzed using a scanning electron microscope, and their crystallographic characteristics were determined using X-ray diffraction, providing a comprehensive understanding of the coating structure. To evaluate the mechanical properties, nanoindentation tests were employed, offering precise measurements of hardness and elasticity. The tribological characteristics of the DLC films were assessed using a pin-on-disc tribometer, examining their wear resistance and frictional behavior under ambient air. These comprehensive analyses reveal the a-C:H:Nb coating potential for applications requiring enhanced surface properties, combining enhanced superior tribological and corrosion performance.

MC-ThP-6 Improving Tribological Properties of Al 7075 Alloy by Two-Step Soft Plasma Electrolytic Oxidation, Thiago de Lima Gontarski (thiago.gontarski@pucpr.edu.br), G. Caetano, J. dos Santos Junior, B. Leandro Pereira, R. Diego Torres, P. Soares, Pontifical Catholic University of Paraná, Brazil

The trend of using aluminum (AI) alloys in various industrial sectors, including naval, automotive, and aerospace, can be attributed mainly to their high specific strength. However, their relatively lower resistance to wear and corrosion could limit their applications. To address this, Plasma Electrolytic Oxidation (PEO) has emerged as an effective method to enhance the mechanical, chemical, and thermal properties of AI alloys. Hence, this study aims to evaluate the impact of sample exposure time during the PEO process on the tribological properties of the AI7075.

Specimens of Al7075 were abraded with silicon carbide sandpapers of #220 grit. Subsequently, all samples were cleansed with acetone and air-dried at ambient temperature. The PEO procedure was carried out in two stages. It employed a unipolar power source, a stainless steel counter electrode, and a silicate-based electrolyte. The first stage of PEO was the same for all samples, and it involved applying a voltage of 300V and a current of 0.5 A for one minute. The second stage was carried out with a voltage of 350V, and a current of 0.3 A, with varied exposure durations for each sample: 3, 5, 10, and 20 min. The morphology, chemical composition, and crystalline phases were characterized using Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), and X-ray Diffraction. The friction and wear properties of the samples were determined by dry linear reciprocating sliding tests in a ball-on-plate setup, using an Anton-Paar universal tribometer. An applied load of 5 N and a sliding speed of 2.5 cm/s were maintained, with a reciprocating stroke of 6 mm. The test distance was set at 40 m at 25°C and a relative humidity of 50%. SEM analyses post-PEO process revealed that surface layers of the Al substrate were characterized by numerous pores and a flattened topography. The smoothest and thickest layer was achieved with the 20-minute PEO treatment. EDS results indicated that Al and O elements were predominantly present in all coatings after various exposure times. The coefficients of friction recorded were 0.557 for the substrate, and 0.501, 0.540, 0.442, and 0.427 for the PEO treatments of 3, 5, 10, and 20 min, respectively. Concurrently, wear rates were measured at 2.84, 1.79, 2.06, 1.97, and 1.34x10<sup>-3</sup>mm<sup>3</sup>/Nm for the same conditions. The oxide layer with the most advantageous tribological performance was that which formed over 20 min: it withstood rupture for in excess of 1600 cycles, compared to the other layers, which failed between 700 to 800 cycles. In conclusion, the longest exposure time during the mild PEO treatment correlated with the most favorable tribological properties.

### MC-ThP-8 Mechanical and Tribological Behavior of Nanolayered Sputtering MoN/MoWN Coatings, W. Hsu, Fan-Bean Wu (fbwu@nuu.edu.tw), Department of Materials Science and Engineering, National United University, Taiwan

This research investigated the microstructure, mechanical and tribological behavior of the molybdenum nitride, MoN, molybdenum tungsten nitride, MoWN, single layers and the nanolayered MoN/MoWN, films through reactive radio frequency magnetron sputtering, RFMS, technique. The nanolayered MoN/MoWN was prepared with fixed 50 nm MoWN building layers and MoN building layers with a thickness of 25 to 50 nm. These layers were alternately stacked to form a multilayer film with a total thickness of approximately 1 µm. The MoN single building layers presented a nanocrystalline structure while well crystalline feature was found for the MoWN layers. Through microstructure analysis, the nanolayered MoN/MoWN with a building bilayer of 25/50 nm/nm possessed continuous growth of MoWN columnar crystals along B1-MoN(111). On the contrary, the through-layer columnar grain was suppressed by the 50/50 nm/nm MoN/MoWN stacking. For mechanical and tribological behavior, the wear track of the M-50/50 multilayer film was shallower and narrower as compared to those of the 25/50 MoN/MoWN multilayer film. The superior wear resistance was attributable to the effective inhibition of continuous growth of columnar crystals by a thicker MoN building layer. Additionally, the 50/50 multilayer MoN/MoWN film exhibited larger compressive residual stress which was beneficial for hardness and tribological characteristics.

### MC-ThP-10 Influence of Carbon and Boron Additions on the Wear Resistance of Fe<sub>3</sub>Al Based Laser Claddings, *H. Rojacz, K. Pichelbauer, M. Varga,* AC2T Research GmbH, Austria; *Paul Heinz Mayrhofer (paul.mayrhofer@tuwien.ac.at),* TU Wien, Institute of Materials Science and Technology, Austria

Strengthened iron aluminides exhibit excellent mechanical properties up to 600°C, and are promising candidates to replace Co-, Cr- and Ni- rich coatings for high temperature wear protection. To improve their hardness, different strengthening mechanisms can be chosen accordingly. For this study, precipitation hardening with carbon and/or boron was used to strengthen Fe<sub>3</sub>Al-based iron aluminides. Carbon and boron were alloyed in the range from 0-20 at.% as well as combined up to 10 at.% each to precipitate carbides, borides and carboborides to show the influence on microstructural evolution, hardness as well as wear resistance. A thorough material analysis of the developed laser claddings materials and the present phases was conducted using scanning electron microscopy, electron backscatter diffraction, hot hardness testing, nanoindentation as well as high temperature abrasion testing. Results show that the hardness can be significantly increased from ~260 HV10 (claddings without any

strengthening of the Fe<sub>3</sub>Al phase) to ~850 HV10 with boride precipitations (20 at.% B). Strengthening with carbon and boron leads to a hardness of ~670 HV10 due to the formation of carboborides as well as graphite islands (10 at.% B and 10at.% C). Alloying with carbon causes the formation of graphite lamellae as well as perovskite-type carbides Fe<sub>3</sub>AlC0.6 and lower hardness of a max. of ~350 HV10 at 20 at.% C. Wear results indicate a strong dependence on the present phases, whereas a significant reduction of the wear rates can be pointed out when strengthened; comparable to classical FeCrC-based hardfacings, but with the advantage of a significantly reduced ecological impact.

#### MC-ThP-11 Understanding Stress in Sputter-Deposited Ti-Zr-N Alloy Films, E. Chason, Tong Su (tong\_su@brown.edu), Z. Rao, School of Engineering, Brown University, USA

Understanding and controlling residual stress in sputtered metal-nitride films is important because of the impact it can have on their properties.Numerous studies have quantified the stress for different systems and processing conditions and modeling has attempted to explain the stress in terms of the underlying kinetic processes.Although ternary nitride alloys are used in many applications, there is much less understanding of stress in these systems relative to the binary alloys.In this work, we present results of stress in TiZrN, TiN and ZrN at different growth rates and pressures.Comparison of the ternary alloy with the two constituent binary alloys sheds light on how the addition of a second metal element modifies the stress. The results are interpreted in terms of mechanisms that have been proposed for explaining the stress generation in sputter-deposited films. These include tensile stress due to island coalescence and compressive stress due to insertion of excess atoms into the grain boundary and the effect of energetic particle bombardment.

#### MC-ThP-12 Cyclic Laser Thermal Shock Resistance and Mechanical Properties of AlCrSiN/AlTiSiN Multilayer Hard Coatings, Ming-Xun Yang (u6au6vmp711@gmail.com), National Formosa University, Taiwan; C. Chang, B. Chang, Y. Chang, National Formosa University, Taiwan

In recent years, the quaternary coatings such as AlCrSiN and AlTiSiN were excellent potential candidates for wear resistance applications due to excellent thermally stability and oxidation resistance. However, for demanding operations such as the interrupted cutting and piercing, a typical failure mechanism of these tools is cyclic thermal fatigue. Such hard coatings may suffer cracking and spalling due to the high temperature cycling impact. A hard coating with multilayer architectures possesses excellent thermal stability and oxidation resistance which is attributed to the multielement composition and multilayer structure. In this study, the cyclic thermal fatigue shock test was developed to investigate the thermal fatigue failure mechanisms of the AlCrSiN and AlTiSiN monolayered coatings and multilayered AlCrSiN/AlTiSiN coatings. The failure mechanisms of the prepared coatings under the constant and cyclic thermal shocks were compared and analyzed. Several instruments were used to analyze the characteristics of the coatings. Emission electron probe microanalyzer (EPMA) was employed for elemental composition analyses, and grazing incidence X-ray diffraction (GIXRD) was utilized to analyze the crystal structure and phases of the coatings. Field emission scanning electron microscopy (FESEM) and field emission transmission electron microscopy (FETEM) were used to observe the cross-sectional microstructures. The coatings were also subjected to scratch tests to determine the adhesion strength of the deposited coatings. A nanoindentation tester was used to measure the hardness and elastic modulus. Ball-on-disk tests were used for the tribological analyses. In this case, ball-on-disk tests were used to evaluate the wear and abrasion resistance of the coatings. The results showed that the AlTiSiN/AlCrSiN multilayer coating exhibited improved mechanical properties and wear resistance due to the multilayer structure. SEM observations of the surface morphology of the thin films after single and 100 cycles of laser irradiation revealed that the AlTiSiN/AlCrSiN coatings showed less laser-induced thermal cracks, indicating excellent thermal fatigue performance of the multilayer coatings compared to respective monolayer coatings.

MC-ThP-13 Fracture Toughness of Borided AISI 1045 Steel with a Diffusion Annealing Process, A. MENESES AMADOR, Instituto Politécnico Nacional, Mexico; A. OCAMPO RAMIREZ, Universidad Veracruzana, Mexico; A. Ballesteros-Arguello, J. Ceron Guerrero, FELIPE NAVA LEANA (felnaval@gmail.com), Instituto Politécnico Nacional, Mexico

A numerical-experimental study of the fracture toughness of iron borides obtained by cross-sectional scratch test was carried out. The iron borides were formed on an AISI 1045 steel. The powder-pack boriding process was developed at 1000 °C and 4 h of exposure time. The diffusion annealing

process was performed on the borided steel at a temperature of 1000 °C with 4.5 h of exposure using SiC powder. The scratch tests were carried out on the cross-sections of borided material using a CSM Revetest-Xpress commercial equipment with a Vickers indenter. The scratch distance was of 1.2 mm with a load range from 5, 10 and 15 N. The applied loads and damage observed at the samples surface (with half cone geometry) were used to estimate the fracture toughness of the system. The numerical model based on the finite element method of the cross-sectional scratch test was developed considering the same test conditions. The numerical results were used to establish parameters employed in the methodology of fracture toughness by cross-sectional scratch testing.

MC-ThP-14 Influence of Cu Addition on Microstructure, Mechanical and Tribological Properties of Fe/NbC Coatings Produced on Tool Steel Using Laser Surface Alloying, Dariusz Bartkowski (dariusz.bartkowski@put.poznan.pl), Poznan University of Technology, Poland; P. JURČI, Slovak University of Technology in Bratislava, Slovakia; A. BARTKOWSKA, Poznan University of Technology, Poland; P. GOGOLA, Slovak University of Technology in Bratislava, Slovakia; D. PRZESTACKI, A. PATALAS, M. ROGALEWICZ, P. POPIELARSKI, P. SIWAK, Poznan University of Technology, Poland

The work presents the influence of manufacturing parameters on properties of Fe/NbC composite coatings metallurgically bonded with tool steel substrate during the laser surface alloying. The Fe/NbC coatings were produced in two stages. In the first stage, an pre-coat in the form of paste was applied on substrate. In the second stage, pre-coat was remelted using a 3 kW diode laser beam. Three laser beam powers: 350 W, 500 W and 650 W were used. Various variants of powder mixture to produce pre-coats were applied: 100% NbC, NbC/4%Cu, NbC/8%Cu and NbC/12%Cu. The amounts of individual components were determined by weight. The main goal of the research was to check the possibility of producing composite coatings reinforced with NbC particles. The influence of copper addition on the properties of these coatings was investigated. Microstructure, microhardness, chemical composition tests using energy-dispersive X-ray spectroscopy and phase composition tests using the X-ray diffraction method were carried out. For tribological tests the Ball-on-Flat sliding wear method was used. The Fe/NbC coatings were analyzed both for adhesion using bronze balls and for abrasion using two types of balls - steel and tungsten carbide. The pressure forces of individual balls were selected in such a way that the average contact stresses in accordance with Hertz's theory were 1 GPa. Basic mechanical properties were determined using the nanoindentation method. It was found that it is possible to produce composite coatings metallurgically bonded to steel substrate, in which the reinforcing phase is NbC, and the role of the matrix is played by iron taken from the substrate or a mixture of iron and copper introduced with the precoat. It was found that some of the NbC particles were melted completely and released in situ in the matrix. However, some of these particles remain in the primary form. It was found that increasing the copper content in precoat leads to decrease in the microhardness of the coating matrix. In the case of coatings without copper, the hardness of 800 HV was achieved, and in the case of the addition of 12% Cu, the maximum hardness was 600 HV. However, this was the hardness of matrix without taking into account the reinforcing particles in the form of primary NbC carbides. Similar relationships occurred in the case of wear resistance. In this study design of experiment methods, which made it possible to determine the significance of the impact of researched input variables of the manufacturing process on the properties of obtained coatings were used.

### MC-ThP-15 Microstructure, Mechanical and Tribological Behavior of Fe/Mo2c Coatings Produced by Laser Surface Alloying on Tool Steel, D. BARTKOWSKI, A. BARTKOWSKA, Poznan University of Technology, Poland; P. JURČI, M. KUSY, Slovak University of Technology in Bratislava, Slovakia; D. PRZESTACKI, Michał ROGALEWICZ (michal.rogalewicz@put.poznan.pl), P. SIWAK, P. POPIELARSKI, Poznan University of Technology, Poland

The work presents the influence of manufacturing parameters on the microstructure and properties of metallurgical Fe/Mo<sub>2</sub>C composite coatings produced in the laser surface alloying process. Tool steel was used as the substrate. The coatings were produced in two stages. In the first stage, precoats based on Mo<sub>2</sub>C powder were produced. Thicknesses of these precoats were 150  $\mu$ m, 250  $\mu$ m and 350  $\mu$ m. In the second stage, pre-coats were remelted with steel substrate using a 3 kW diode laser beam. A constant laser beam scanning speed of 3 m/min and three laser beam powers: 500 W, 700 W and 900 Wwere used. The aim of the studies was to determine the possibility of producing coatings with a composite microstructure, where the matrix will consist of iron from the substrate and Mo<sub>2</sub>C particles will be the reinforcing phase. Microstructure tests using a

scanning electron microscope, microhardness tests, chemical composition tests using the energy-dispersive X-ray spectroscopy method and phase composition tests using the X-ray diffraction method were carried out. Basic mechanical properties were checked using nanoindentation. Wear resistance tests were also carried out. To determine friction properties Amsler type method was used. Hardened steel was used as a counter specimen. It was found that it is possible to produce composite coatings metallurgically bonded to the substrate, in which the reinforcing phase is Mo<sub>2</sub>C and the role of the matrix is played by iron from the steel substrate. This work presents the model for producing this type of coatings. It was found that the laser beam power and the thickness of pre-coats have influence on obtained Fe/Mo<sub>2</sub>C coatings properties. Thanks to the use of design of experiments methods, the significance of the influence of the tested production parameters on the properties of the obtained coatings was determined. It was found that increasing the laser beam power leads to a decrease in the hardness of Fe/ Mo<sub>2</sub>C coatings. The most favorable hardness and wear resistance are obtained for coatings produced using precoat thickness of 150 µm and 250 µm. Further increasing the thickness of the pre-coat results in deterioration of mechanical and operational properties. This is related to the reduced amount of matrix material in the coating and thus the lack of a matrix binding the Mo<sub>2</sub>C particles.

MC-ThP-16 Mechanical Properties, Microstructure and Tribological Behavior of TaC Coatings Produced Using Laser Surface Alloying on Monel®400 Alloy, A. BARTKOWSKA, D. BARTKOWSKI, Poznan University of Technology, Poland; P. JURČI, Slovak University of Technology in Bratislava, Slovakia; D. PRZESTACKI, Paweł POPIELARSKI (pawel.popielarski@put.poznan.pl), P. SIWAK, A. MIKLASZEWSKI, M. ROGALEWICZ, Poznan University of Technology, Poland

The work presents the characteristics of metallurgical TaC coatings produced on the single-phase Monel®400 alloy using the laser surface alloying method. The coatings were produced in two stages. In the first stage, pre-coats based on TaC powder were produced. They had a thickness of 260  $\mu$ m +/- 10  $\mu$ m. In the second stage, pre-coats were remelted with a Monel®400 alloy substrate using a 3 kW diode laser beam. A constant scanning speed of laser beam: 3 m/min and three laser beam powers: 350 W, 450 W and 550 W were used. Microstructure tests using a scanning electron microscope and microhardness tests were carried out. Additionally chemical composition tests using the Energy-dispersive X-ray spectroscopy method and phase composition study X-ray diffraction were investigated. The basic mechanical properties of the produced coatings were determined using nanoindentation. Tribological tests were also carried out. Test method for linearly reciprocating Ball-on-Flat sliding wear was used. The produced coatings had a composite microstructure with a clearly separated TaC reinforcing phase from the nickel-copper based alloy matrix. It was found that it is possible to create composite coatings on the Monel®400 alloy while simultaneously implementing this alloy from the substrate to the coating as a matrix. Based on the conducted studies, it was found that the increase in the laser beam power has influence on decrease of content of the reinforcing phase. The microstructure influences both microhardness results and wear resistance. In the matrix area microhardness ranging from 400 HV to 700 HV were obtained, but much higher values (even over 1600 HV) were observed in the areas significantly changed by the melted TaC carbide. The significance of influence of laser beam power on individual mechanical properties was determined. It was taken into account that increasing the power of the laser beam contributed to a change in the microstructure, including the complete melting of some primary TaC particles and their separation as secondary carbides.

MC-ThP-17 Influence of Differently Manufactured TiAl Targets on the Structural and Tribo-Mechanical Properties of Arc-Evaporated TiAlN Thin Films, Finn Ontrup (finn.ontrup@tu-dortmund.de), N. Lopes Dias, TU Dortmund University, Germany; D. Stangier, Oerlikon Balzers Coating Germany GmbH, Germany; N. Denkmann, A. Meijer, S. Jaquet, J. Debus, D. Biermann, W. Tillmann, TU Dortmund University, Germany

The cutting performance and service life of WC-Co milling tools can be improved through the application of TiAlN thin films. Among the different Physical Vapor Deposition (PVD) methods, arc evaporation is widely employed to coat cutting tools due to its high deposition rate and excellent adhesion of the thin films. The TiAl targets are typically produced either by smelting or powder metallurgical methods, depending on the Al/Ti ratio. Smelting is commonly employed for Al/Ti ratios up to 1, while powder metallurgy becomes necessary for Al/Ti ratios exceeding 1. The impact of different deposition parameters on the arc evaporation process is well studied, while little is known about the influence of the target manufacturing route on the resulting tribo-mechanical properties.

Differences in the manufacturing route manifest themselves in the microstructure of the targets, which in turn results in different arc conditions at the target surface during deposition and may also cause a droplet formation. Therefore, TiAlN thin films are deposited by advanced plasma assisted (APA) arc sources, using smelting and powder metallurgical TiAl targets at three distinct working pressures (8500, 5000 and 3000 mPa).

The correlation between the target and the deposition conditions is analyzed with respect to quantity and type of droplets within the thin film. These measurements are supplemented by 3D optical roughness measurements of the thin film surface. Scanning electron microscopy SEM is used to analyze the morphology and topology, while the chemical composition is determined by microprobe analysis as well as tip-enhanced Raman scattering. Additionally, the influence of the different target types on their phase composition is evaluated employing X-ray diffraction. The hardness and the coefficient of friction are determined to examine the tribo-mechanical behavior of the thin films on cemented carbide substrates. These comprehensive analyses provide insights into the relationship between the target manufacturing route and the resulting structural, physico-chemical and tribo-mechanical properties of TiAIN thin films. The results will enhance the fundamental understanding of the interaction between target type and thin film properties of arc-evaporated TiAIN.

### MC-ThP-18 Formation of TiB<sub>2</sub>/TiB Layers on Ti<sub>6</sub>Al<sub>4</sub>V Alloy: Adhesion and Wear Resistance, J. Escobar-Hernández, G. Rodríguez-Castro, J. López-Rodríguez, A. Meneses-Amador, A. Cruz-Ramírez, T. N. Cabrera-Yacuta (tcabreray1800@alumno.ipn.mx), Instituto Politécnico Nacional, Mexico

Adhesion and wear of Titanium borides (TiB<sub>2</sub> and TiB) formed on Ti<sub>6</sub>Al<sub>4</sub>V alloy wereevaluated by scratch tests. The powder-pack boriding at 1100 °C during 10, 15 and 20 h under inert argon atmosphere was applied to Ti6Al4V alloy. TiB2 and TiB phases were identified on the surface of Ti alloy with maximum thicknesses of 9.3 and 8.6 µm, respectively. By instrumented indentation, hardnesses were determined around 42 GPa for TiB2 and 24 GPa for TiB. The Rockwell C tests classify the adhesion of the systems as acceptable (HF3 type), regardless of the layer thicknesses. While in the scratch tests, the behavior of the coefficient of friction increased from 0.2 to 0.6 as the indenter penetrates and the damage mechanisms identified were hertzian cracks, chipping, and spallation. After, the multipass scratch test was employed to evaluate wear behavior at subcritical loads (40 and 50% of chipping critical load) applying 100 cycles. According to the results, the friction coefficient was not affected by the titanium boride thicknesses, but the wear rate reached its maximum reduction at 20 h of boriding with 3,7 x10<sup>-3</sup>mm<sup>3</sup>/N\*m.

### MC-ThP-19 Effect of MoS<sub>2</sub> Additive on Corrosion and Tribocorrosion Property of Plasma Electrolytic Oxidation Coating on Titanium, N. Zheng, National Taiwan University of Science and Technology, Taiwan; *Chun-Wei Chang (yiwenz988@gmail.com)*, Ming Chi University of Technology, Taiwan, Republic of China; *C. Wang*, National Taiwan University of Science and Technology, Taiwan; *C. Tseng*, Ming Chi University of Technology, Taiwan, Republic of China

Plasma electrolytic oxidation (PEO) technology as a novel and attractive surface engineering process has been widely used for preparation of functional oxide coatings on light alloys such as aluminum, magnesium, zirconium, and titanium. In this study, we fabricated the MoS<sub>2</sub> decorated composite oxide layers on pure titanium by using PEO treatment under pulsed DC power with unipolar mode in alkaline phosphate- and aluminatebased solutions with 0~3 g/L MoS<sub>2</sub> nanoparticle additions. The influence of MoS<sub>2</sub> nanoparticle addition on the microstructure, mechanical property, corrosion resistance and tribocorrosion behavior of PEO composite coating on pure titanium was investigated by X-ray diffraction (XRD), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), field-emission electron probe microanalysis (FE-EPMA), surface profilometry (a-step), scratch adhesion testing, pin-on-disc wear testing and potentiodynamic polarization measurement in 3.5 wt% NaCl solution. The experimental results obtained from scratch adhesion testing and potentiodynamic polarization measurements show that PEO composite coating with 2.5 g/L  $MoS_2$  nanoparticles addition exhibits optimal adhesion strength and corrosion resistance. Furthermore, the results of XRD and SEM-EDS indicate that regardless of the presence or absence of MoS<sub>2</sub> nanoparticle additives, the PEO composite coatings on pure titanium are primarily composed of aluminum titanate (Al<sub>2</sub>TiO<sub>5</sub>) and rutile-phase titanium dioxide (TiO<sub>2</sub>). The FE-EPMA data reveal that MoS<sub>2</sub> particles are mainly well distributed at the interface between the PEO coating and pure titanium substrate. The tribocorrosion behavior of MoS2 nanoparticle

decorated PEO composite coatings was carried out by potentiodynamic polarization measurement in 3.5 wt% NaCl solution under wear mode. As similar to static potentiodynamic polarization measurement, the PEO composite coating with 2.5 g/L MoS<sub>2</sub> nanoparticles addition also displays optimal tribocorrosion resistance in this study. In summary, the adhesion strength, wear resistance and corrosion/tribocorrosion resistance of Al<sub>2</sub>TiO<sub>5</sub>-rutile TiO<sub>2</sub> composite coating on pure titanium can be improved by increasing MoS<sub>2</sub> nanoparticles addition. The optimal concentration of MoS<sub>2</sub> additive is 2.5 g/L.

#### MC-ThP-20 An Improved Statistical Nanoindentation Methodology, Esteban Broitman (esteban.daniel.broitman@skf.com), Y. Kadin, P. Andric, SKF - Research and Technology Development, Netherlands

The principle of statistical nanoindentation proposed ca. 2006 is based on performing a relatively large number (many hundreds/few thousands) of single indentation tests in a grid and analyzing the indentation elastic modulus and hardness with statistical methods. Many authors have claimed that this method can be used to study composite materials, showing that mechanical properties, number, and volume of the different composite phases could eventually be deduced and predicted by only using the nanoindentation technique.

In this presentation, we first review the previous work done in statistical nanoindentation by different researchers, highlighting the main problems that have been encountered and possible proposed solutions. In the second part, we study and report the statistical nanoindentation of three composite model samples, in the form of a soft Al2124 matrix embedded with hard SiC particles. We propose a novel heuristic wavelet technique to filter the measurement noise from the raw nanoindentation data as an attempt to obtain a more robust statistical nanoindentation methodology. Furthermore, a Finite Elements modeling will be used to analyze the response of the nanoindenter regarding the position of the hard particles. Our modeling will show many mistakes made by authors in previous publications. Finally, we will introduce results on bearing steels. Hardness histograms generated by Statistical Nanoindentation will demonstrate unique characteristics (fingerprints) for the different analyzed steels.

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MC-ThP-21 Adhesive Strength and Diffusion Model for Borided Ti6Al4V Alloy, A. MENESES AMADOR, G. RODRIGUEZ CASTRO, Instituto Politécnico Nacional, Mexico; DIEGO ALONSO BAUTISTA ALVAREZ (d.bautistaalvarez@gmail.com), INSTITUTO POLITECNICO NACIONAL, Mexico; I. CAMPOS SILVA, Instituto Politécnico Nacional, Mexico

In this work, the Ti6Al4V alloy was hardened by powder-pack boriding process at temperatures from 1000 to 1100 °C for 10. 15 and 20 h. The boride layers formed on the Ti6Al4V alloy were examined by scanning electron microscopy and X-ray diffraction, while their mechanical behavior was evaluated by Berkovich nanoindentation and Hertzian contact. The growth kinetics of the boride layer formed on Ti6AL4V alloy was investigated based on the boron activation energy. The boride layer consisted of an outer TiB2 layer and a TiB whiskers sub-layer. A diffusion model was proposed to estimate the boron activation energies in the TiB<sub>2</sub> and TiB layers, where values of 91.2 and 146.5 kJ mol<sup>-1</sup> were obtained considering the experimental data of the thicknesses of the boride layers. Young's modulus and hardness were at the range of 350-400 GPa and 20-25 GPa for TiB and TiB<sub>2</sub> phases, respectively. The sample with the thinnest layer thickness showed the highest adhesive strength under Hertzian contact. Finally, finite element method was used to obtain the stress field in the layer-substrate system caused by the contact loads.

MC-ThP-22 Influence of Ti Content on the Tribological Behavior of Ti:MoS<sub>2</sub> Coatings Under Reciprocating Electrified Contact Conditions, N.K. Fukumasu (newton.fukumasu@usp.br), M. Danelon, A. Tschiptschin, I. Machado, R. Souza, University of São Paulo, Brazil

The possibility of improving the durability and efficiency of heavy-loaded mechanical transmission systems by controlling external parameters is paramount in next-generation automotive powertrain and energy generation systems. Using electric current to control surface chemistry during the relative motion between two surfaces could allow electrified contacts to present reduced friction and wear, reducing energy consumption and enhancing overall system performance. The use of coatings with advanced 2D materials, such as molybdenum disulfide, may promote excellent solid lubrication under high contact stresses and pure sliding conditions. In this work, MoS2coatings were deposited using pulsed D.C. magnetron sputtering technique with MoS<sub>2</sub> (purity of 99.99%) and Ti (purity of 99.9%) targets, in which changes in the power applied to the Ti target allowed the variation of the final Ti doping concentration at Ti:MoS<sub>2</sub> composite coatings. Reciprocating electrified tribological tests were carried out with the ball-on-plane configuration. Three electrified conditions (nonelectrified, ball as cathode and ball as anode) and five Ti doping concentrations were tested. In all cases, uncoated AISI 52100 balls were pressed with a 10 N normal load against Ti:MoS<sub>2</sub> coated glass substrates. Tangential ball velocity was set at 3 mm/s with 4 mm stroke movement distance. Results indicated that electrification of the contact induced lower COF than the non-electrified conditions, for coatings with lower Ti concentration. Raman Spectroscopy of inner regions of the wear tracks indicated the presence of crystalline  $MoS_2$  compared to as-deposited coatings. Coherence correlation interferometry analyses of wear tracks indicated wider tracks for the cases in which the ball was set as the cathode of the system. Also, under this condition, optical and scanning electron microscopy results showed high coating damage for all tested Ti concentrations. Results suggest that electrical potential polarity may promote selective desorption of coating ions that change surface chemistry, influencing the formation and composition of tribofilms that develop during the sliding motion and the friction and wear behavior of the tribosystem.

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