

## Surface Engineering - Applied Research and Industrial Applications

### Room Sunset - Session G4

#### Pre-/Post-Treatment and Duplex Technology

**Moderators:** Hiroshi Tamagaki, NIRO (The New Industry Research Organization), Wan-Yu Wu, Da-Yeh University, Chris Stoessel, Eastman Chemical Company, Inc., USA

10:00am **G4-1 Mechanical Pretreatment before Electroplating of Aluminium Alloy AlSi12**, *E Uhlmann, Robert Jaczowski*, Technische Universität Berlin, Germany

Electroplating is a coating process which is used to increase the durability of workpieces or to change their appearance. To form a high adhesive bonding between the coating and the workpiece, its surface has to meet certain requirements achieved by a comprehensive pretreatment. The conventional pretreatment process chain consists of different steps to increase the roughness and the surface tension of the workpieces. For this process chain chemicals are used, which are harmful to the environment and health, besides their high costs.

The complexity of the pretreatment and coating process chain differs depending on the used workpiece material. Particularly challenging to coat are Aluminium-Silicon alloys. During the conventional pretreatment of these casting alloys, the Silicon phase enriches on the surface of the workpieces and has to be removed in an additional process step using nitric acid.

This study compares alternative mechanical pretreatment processes for AlSi12-workpieces prior to electroplating. The investigated processes are conventional grinding, double face grinding with planetary kinematics, lapping and dry ice blasting. Subsequent to the structuring, the workpieces are cleaned by carbon dioxide snow blasting. The investigation of the different surface structures and their properties, like roughness, surface tension and wetting behaviour, allowed the determination of their impact on the adhesive strength of the coating by empirical modelling.

By structuring the surfaces of the workpieces they could be coated by electroplating. As a result, the chemicals used in the conventional pretreatment could be substituted. Especially the creation of surfaces, which could be wetted homogeneously, showed a satisfactory adhesive strength of the applied coatings. Concluding the study, a comparison of the economic performance of the mechanical and conventional pretreatment processes is created, which confirms the profitability of the mechanical pretreatment for small batch sizes.

10:20am **G4-2 Microstructure Characterization and Mechanical Properties of Gradient AlCrSiN hard Coatings Using Ternary Alloy Targets**, *Y Chang, Liang-Chan Chao*, National Formosa University, Taiwan

Transition metal nitride coatings based on Cr and Al, such as CrN and CrAlN have been attracting great interest for industrial applications as protective coating materials due to their high hardness, impact resistance and thermal stability. CrAlN coatings show high hardness and high thermal stability together with excellent oxidation resistance. However, the properties of the CrAlN coatings may be further improved by addition of Si. The addition of Si suppresses the grain growth and refines it. In this study, AlCrN and AlCrSiN coatings were deposited onto high-speed steels and tungsten carbide tools using AlCr and ternary AlCrSi alloy targets in a Cathodic-arc evaporation (CAE) system. During the coating process of gradient AlCrSiN, CrN and AlCrN were deposited as interlayers to enhance adhesion strength between the coatings and substrates. By controlling the different negative bias voltages and cathode currents, the AlCrSiN via compositional grading and plasma etching possessed different microstructures and mechanical properties. The microstructure of the deposited coatings were investigated by field emission scanning electron microscope (FE-SEM) and field emission gun high resolution transmission electron microscope (FEG-HRTEM), equipped with an energy-dispersive x-ray analysis spectrometer (EDS), Glancing angle X-ray diffraction was used to characterize the microstructure and phase identification of the films. Mechanical properties, such as the hardness and young's modulus, were measured by means of nanoindentation. The adhesion strength of the coatings was evaluated by a standard Rockwell indentation test. A ball-on-disc wear test was conducted to evaluate the tribological properties of the deposited coatings. In order to evaluate the impact fatigue behavior of the coated samples, an impact test was performed using a cyclic loading device with a tungsten carbide indenter as an impact probe. The design of AlCrSiN coatings were

Monday Morning, April 23, 2018

anticipated to increase the hardness, toughness, thermal stability and impact resistance by optimizing the coating architecture.

10:40am **G4-3 Integrated Shot Peening, Plasma Nitriding and Gradient PVD TiAlSiN Coating on AISI H13 Molds for Al Die Casting**, *Venice Mascariñas*, University of the Philippines, Philippines; *D Quinto*, Beta Nanocoating Philippines Inc., Philippines; *A Salvador*, University of the Philippines, Philippines

Surface engineering that combines processes of a) external shot peening b) plasma nitriding and c) PVD coating – b) and c) comprising duplex treatment done in a continuous process in a commercial PVD cathodic arc machine – has proven to significantly improve the service lifetime of AISI H13 steel molds used for aluminum die casting. Microhardness profiles measured across a spherical taper section (calotte scar) gave a hardness of 2900 HV<sub>0.05</sub> for the 5 μm TiAlSiN-based multilayer coating while the hardness of the substrate gradually decreased from 1300 HV<sub>0.05</sub> at the surface across the 40 μm nitrided depth to 600 HV<sub>0.05</sub> of the base steel. XRD, SEM-EDS analyses confirmed the presence of Fe<sub>2-3</sub>N and CrN phases within the nitrided diffusion layer of the H13 steel. Shot peening effected by alumina particle microblasting resulted in enhanced nitriding. Rockwell indentation tests at different loads were utilized to compare the adhesion of TiAlSiN coating on the plasma nitrided and non-nitrided H13 samples. Coating removal along circumferential cracks was observed on the non-nitrided sample at 100 kg and 150 kg loads compared to no coating removal on the duplex-treated H13 steel at these loads. The mechanisms of performance improvement of duplex treated H13 molds subjected to thermal fatigue and tribological wear can thus be attributed to the synergies of 1) a high-temperature wear-resistant hard coating, 2) a nitrided layer of increased hardness and in compressive residual stress that inhibits cracking and gives higher load support to the coating against plastic deformation and delamination, and 3) prior shot-peening that enhances nitrogen diffusion during plasma nitriding.

11:00am **G4-4 Effect of Nano-penning Surface Texturing on Self-clean Function**, *Nicolas Coniglio*, Arts et Métiers ParisTech d'Aix-en-Provence, Laboratory of Mechanics, Surface and Materials Processing (MSMP-EA7350), France; *S Mezghani*, Arts et Métiers ParisTech de Châlons-en-Champagne, Laboratory of Mechanics, Surface and Materials Processing (MSMP-EA7350), France; *M El Mansori*, Arts et Métiers ParisTech d'Aix en Provence, Laboratory of Mechanics, Surface and Materials Processing (MSMP-EA7350), France; *J Cabrero*, Saint Gobain, CREE, France

Surface texturation at micro and meso scales play an important role in applications where cosmetic, aesthetic and clean functionalities are specified. We are hence dealing with a multiscale surface, in which texturing and texture have a larger influence because they are scaled differently. In this research paper, an experimental method is illustrated to highlight the important effect on the anti-fingerprinting performance (i.e. surface hydrophobicity) rated in term of surface wettability. We examine first, in detail, the wetting response of surfaces textured on aluminum alloy 6063 plates using nano-peening with a range of processing parameters. Roughness was measured by atomic force microscopy (AFM) over a 100 x 100 μm<sup>2</sup> surface. In addition, the surface wettability was quantified by measuring the contact angles of different liquids using the sessile drop method according to the norm AFNOR EN 828. The calculation takes into account the wetting behavior of the textured surfaces at different scales. A correlation was made between the micro-scale roughness and the macro-scale apparent solid surface energy.

11:20am **G4-5 Hard Coating and Surface Modification Technologies for Piston Ring**, *Hideaki Kamiyama*, Nippon Piston Ring Co., Ltd., Japan

INVITED

The technology that achieves abrasion resistance and low friction is essential for improving the product features and the added value of slide members, which are used under severe conditions such as in a high-temperature and high-speed environment. Typically, abrasion resistance and low friction are achieved with the use of the deposition technique, which involves coating of a material surface with a hard coating, and/or the modification technique, which hardens the material surface itself.

A piston ring is an engine component fitted into the groove cut in a piston (piston groove), and moves in a reciprocating motion with the piston inside a cylinder. Specifically, a piston ring has two different surfaces: a surface that contacts the cylinder (peripheral surface), and a surface that contacts the piston (slide faces, or inside surface). These surfaces contact different materials, and different functions are required for these surfaces. It is accordingly very important to have possession of not one but many surface treatment techniques.

# Monday Morning, April 23, 2018

The deposition technique used for piston rings often uses chrome plating by a wet process, and CrN, and TiN coating by a dry process (Arc Ion Plating, AIP). Gas nitriding and salt bath nitriding are two processes commonly used for surface modification. Though the deposition and surface modification techniques are often used by themselves, these techniques are also used in combination depending on engine specifications.

In this lecture, we introduce a technology based on both deposition and surface modification, along with the recent composite technology.

## Surface Engineering - Applied Research and Industrial Applications

### Room Sunset - Session G3

#### Innovative Surface Engineering for Advanced Cutting and Forming Tool Applications

**Moderators:** Heidrun Klostermann, Fraunhofer FEP, Holger Gerdes, Fraunhofer Institute for Surface Engineering and Thin Films IST, Mirjam Arndt, OC Oerlikon Balzers AG, Liechtenstein

**1:30pm G3-1 On the Synergies Between Coating and Tool Material Substrate: A Strategy to Optimize Coated Tools Performance in Cold Forming.** *D Casellas*, Fundació CTM Centre Tecnològic, Spain; *A Mueller*, Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein; *Giselle Ramirez*, *M Vilaseca*, Fundació CTM Centre Tecnològic, Spain **INVITED**

In recent years hard coatings have acquired a significant importance in the microstructural and micromechanical design of tools and components. Among the wide range of surface modification techniques, PVD (Physical Vapour Deposition) is one of the most suitable routes to meet the demanding requirements of surface finish and dimensional tolerances that need some specific tools and components, such as those for the automotive sector. However, although thin films tend to reduce the wear mechanisms under service conditions, improved resistance against contact fatigue failure still remains a challenge. The latter has been emphasized in punching and forming processes, where the introduction of advanced materials (such as high strength steels, thick stainless steel strips, etc.) requires higher impact pressures than those used in conventional materials. In those cases, the load is mainly supported by the substrate. Thus, if any subsurface damage occurs in coated tools, then a premature detachment of the layer, or even more, chipping of material could be produced accelerating the wear damage mechanism.

Attempting to ensure the effective usage of coated steels under complex service conditions, where wear is accompanied by contact loads of cyclic nature, it is important to understand and document the interaction between coating and substrate of industrial components. Once the main mechanism of damage is identified on cold forming tools, the following step is the selection of the optimum configuration of coating, interface and steel. But the selection process is not trivial task, and a wrong decision can produce bad experience in the application of coating technology. Recent studies have demonstrated that topography and mechanical properties of the PVD coated surfaces is strongly dependent on the tool steel microstructure as a substrate. This was mainly attributed to a difference in sputtering rates of phases and the metal-matrix of steels during ion etching process, and also due to the size, morphology and structure type of primary carbides.

The main objective of this work is to address the interaction between tool steel and thin coating during cold forming and discern the most relevant microstructural and topographical aspects leading to high tool performance. Detailed microstructural analysis will be complemented with industrial tool behavior to properly understand the synergies between coating and tool material substrate.

**2:10pm G3-3 Deposition of ta-C Coating by Arc Ion Plating for Machining of Al Alloys.** *Yoshiyuki Isomura*, *T Takahashi*, *S Kujime*, Kobe Steel, Ltd., Japan

Al-alloy is soft metallic material and exhibits good machinability. However welding adhesion and building-up edge (BUE) tend to form easily at a surface of cutting tool due to its low melting point. To prevent the formation of BUE, dry cutting tool for Al-alloy requires high resistance against welding adhesion. For this purpose Diamond-Like Carbon, DLC, coating draws a great practical attention. DLC is known to exhibit low friction and high resistance to welding adhesion. Among various types of DLC coatings practically available in recent years, hydrogen free DLC, also referred to as ta-C, tetrahedral amorphous carbon, shows high sp<sup>3</sup> content and hence high hardness as compared to conventional hydrogen containing DLC coatings.

In this study, we deposited ta-C coatings by cathodic arc process, also referred to as arc ion plating, in a middle sized industrial coating unit. The unit was equipped with a round-bar type cathode with a typical target diameter of 20 mm. Pure graphite was used as the target material. ta-C coatings were deposited at different bias voltage and film thickness. The sp<sup>3</sup>/sp<sup>2</sup> fraction, hydrogen content, and film density were studied by XPS,

RBS, and XRR method, respectively. These coatings were also deposited on cutting tools for machining of Al alloys. Correlation of the film properties to cutting performance is discussed.

**2:30pm G3-4 Laser Structured High Performance PVD Coatings for Injection Molds.** *K Bobzin*, *T Brögelmann*, *N Kruppe*, *Mona Naderi*, Surface Engineering Institute - RWTH Aachen University, Germany

Structured plastic components, which are used for flow-optimization, self-cleaning surfaces and optical applications, are usually produced by injection molding using structured tools. A suitable technique for structuring is laser ablation, which offers a high level of accuracy and flexibility. Moreover, the dimension of structures fabricated by laser in the order of a few micro- and nano-meters can significantly change surface properties such as topography, mechanical properties and wetting. Specifically, adhesive and abrasive wear of the mold surface are important failure mechanisms in plastics processing. The key to overcome the aforementioned challenges is to modify the surface of the mold using innovative surface engineering such as physical vapor deposition (PVD). However, coating deposition on nanostructures leads to a decisive change in the surface topography and the surface functionality. For this reason a direct structuring of PVD coated tools is beneficial.

This work deals with the influence of laser structuring on the properties of nitride and oxinitride chromium-based coatings. For this purpose, three coatings with different chemical composition were synthesized on tool steel AISI 420 (X42Cr13, 1.2083) by means of high power pulsed magnetron sputtering process. The coatings were structured by means of ultra-short pulsed laser under variation of the parameters, such as pulse energy, repetition rate of laser and structuring speed. It could be observed that a decrease of the laser intensity results in a reduction in the structural depth observed with scanning electron microscopy. This, on the other hand, leads to a decrease of the surface roughness measured through confocal laser scanning microscopy. Structural changes at the surface of the coating as a result of thermal effects during laser structuring were characterized using Raman spectroscopy and in-situ high-temperature X-ray diffraction.

Based on the results of the laser-surface interaction one set of laser parameter was selected. With these parameters, the samples were structured over a large area for the tribological investigations. On these samples, friction behavior was measured by using a pin on disc tribometer in contact with polycarbonate and polymethyl methacrylate. Furthermore, the wetting behavior of plastics melts on the structured coatings was investigated by means of high temperature contact angle measurements. The shear energy while shearing the solidified plastics from the surface was determined. The results for the structured coatings are promising with regard to the production of optical plastics components.

**2:50pm G3-5 Effect of Layer Sequence on Wear Behavior of AlTiSiN Hard Coatings.** *Joern Kohlscheen*, *C Bareiss*, Kennametal GmbH, Germany; *C Charlton*, *D Banerjee*, Kennametal Inc., USA

The aim of this study is to understand the effect of different silicon contents and layer sequences on the resulting wear behavior of AlTiN based coatings. Deposition was done in an industrial scale PVD unit using cathodic arc evaporation of AlTi and TiSi materials. Silicon contents ranging from 3 to 15 at. % (metal fraction of the nitride compound) were adjusted. Monolithic and multilayers were deposited keeping the coating thickness between 3 to 4 micron. Coating hardness and elastic modulus were determined by nanoindentation. Adhesion and toughness were characterized by indentation with a diamond tip. Film structure and chemistry was determined using EDX and XRD. In some cases TEM images were prepared to clarify film structure in the nanometer range. Using fatigue testing at room temperature and T ~ 700 C it could be shown that multi-layers tend to be more wear resistant especially for moderate silicon contents below 10 at. %. In order to tailor the wear resistance in the contact zone of a cutting tool, we varied the silicon content and selectively removed the outer layer to obtain harder and tougher zones at the flank and rake face of carbide inserts (ISO K grade). Turning of high strength cast iron material was performed to show the beneficial effect of this method. The resulting wear patterns will be discussed.

**3:10pm G3-6 Structural, Mechanical, and Cutting Properties of AlCrN Coatings Deposited by Arc Ion Plating.** *N Ohba*, *T Takahashi*, *Susumu Kujime*, Kobe Steel, Ltd., Japan

High aluminum containing transition metal nitride such as AlCrN and AlTiN has widely been used for cutting tool application due to their high hardness and oxidation resistance at elevated temperatures. The intrinsic properties of the coatings, and hence cutting performance thereof is highly dependent

# Monday Afternoon, April 23, 2018

on Al content and its crystallographic structure. It is of practically significance to increase Al content in the coating in a vacuum deposition process whereas the metastable cubic phase is preferred to be remained and the possible formation of AlN hexagonal phase should be suppressed during deposition as well as cutting operational condition.

In this study we investigate the correlation between deposition conditions, coating properties, and cutting performance. AlCrN coatings were deposited by cathodic arc, also referred to as arc ion plating. As for coating process we employed new industrial arc ion plating system having a reasonably compact batch volume for production, but yet high productivity. Al content  $x$  in  $Al_xCr_{1-x}N$  was systematically varied by changing the target composition from 65 up to 73 at.% Al. Bias voltage of 40, 70, and 150 V were applied at a given composition of Al. The structural, compositional, and mechanical properties of the coatings were studied by X-ray diffraction, SEM-EDX, as well as nanoindentation. Residual stress of the coating was also evaluated from curvature measurement of a plate sample. For cutting test evaluation the depositions were also performed on micro-endmills with a diameter of 1 mm. Cutting performance with respect to the coating properties will be discussed.

3:30pm **G3-7 Physical Properties and Cutting Performances Relation to Coating Conditions of AlCrN Coating Deposited by HiPIMS and Cathodic Arc**, *Keizo Tanaka, S Imamura, M Setoyama, H Fukui*, Sumitomo Electric Hardmetal Corp., Japan

Recently Aluminum Chromium Nitride (AlCrN) coating is widely used in cutting tool industry. And HiPIMS technology has been seen as a probable alternative to the cathodic arc technology. In this study, physical properties and cutting performances was examined and the results explained in terms of coating conditions compared HiPIMS to cathodic arc technology.

HiPIMS coating showed valuable surface morphology, hardness ; 22.9 to 41.6GPa, compressive residual stress; 0.3 to 3.3GPa by bias voltage ; 30 to 100V, peak pulse power ; 30 to 60kW and nitrogen partial pressure.

HiPIMS coating showed higher hardness and more stable tool damage than cathodic arc by higher bias voltage or higher peak pulse power. These results were considered argon ion bombardment assist during film deposition. This consideration indicates further usability of HiPIMS technology in cutting tool industry.

4:50pm **G3-11 Nanoscale Multilayer PVD Coatings to Serve in Demanding Environments**, *Papken Hovsepian, A Ehasarian*, Sheffield Hallam University, UK

**INVITED**

Superlattices were discovered in 1925 by Johansson and Linde as periodic structures of layers of two (or more) materials. In 1970 J.S. Koehler theoretically predicted that if these materials were selected to be with high and low elastic constants a super strong man made material could be produced. Various theories and models have been put forward to explain the super hardening and super toughening effects and a large variety of coatings have been explored both on laboratory and industrial scale. This work summarises results on the properties and performance of novel nanoscale multilayer structured coatings produced by High Power Impulse Magnetron Sputtering, (HiPIMS) dedicated to serve in demanding environments.

To protect Gamma-TiAl components used in automotive and aero engines against environmental attack dense oxide forming elements such as Cr and Al were combined to produce **CrAlYN/CrN** nanoscale multilayer coatings. Coatings with very low layer waviness and strongly improved density have been successfully grown by HiPIMS. These coatings provide excellent oxidation resistance up to 850°C and reduce the fatigue deficit of the aerospace turbine blade material to less than 9%.

**TiAlCN/VCN** represents a new class of superlattice coatings where enhanced performance is achieved by lateral segregation of small atom material (in this case Carbon) at the interfaces between the individual layers producing low shear strength interfaces. These coatings provide excellent tool protection against build-up edge formation during machining of Aluminium alloys, Titanium alloys and MMCs widely used in aerospace and automotive applications.

In **Me doped Carbon films** a unique nanoscale multilayer structure was produced by unconventional method of coating growth based on dynamic segregation driven by intensive ion irradiation. By varying the ion energy and ionisation degree, layered structures with bi-layer period of up to 25 nm were grown. With Cr/C and Mo-W doped Carbon nanoscale multilayer films enhanced tribological performance in boundary lubricated conditions at elevated temperatures, (up to 200°C) was achieved. Smart material selection allowed *in-situ* formation of lubricious phases at the asperity

contacts due to tribochemical reactions between the dopants and the oil and improved the coatings high temperature stability.

**CrN/NbN** combining the electrochemically stable Nb with the wear resistant Cr was initially developed as a replacement of hard Cr, however its unique properties allowed the coating to be used in many other demanding applications described here.

**CrN/NbN** provided reliable protection against high pressure, (50 bar) , high temperature, (600°C) pure steam attack on P92 steel used in steam turbines and showed high resistance against water droplet erosion. Furthermore, the coating did not deteriorate the mechanical properties such as Ultimate Tensile, Low Cycle Fatigue and Creep Strength, which is of paramount importance in turbine blade applications.

The performance of medical implants was enhanced by the application of **CrN/NbN** coatings. Metal ion release studies showed a reduction in Co, Cr and Mo release at physiological and elevated temperatures to undetectable levels (<1 ppb). Thorough *in vitro* biological, cytotoxicity, genotoxicity and sensitisation testing proved the safety of the coating in biological environment.

## Surface Engineering - Applied Research and Industrial Applications

### Room Sunset - Session G2

#### Component Coatings for Automotive, Aerospace, Medical, and Manufacturing Applications

**Moderators:** Osman Levent Eryilmaz, Argonne National Laboratory, USA, Jolanta Klemberg-Sapieha, Polytechnique Montréal

8:00am **G2-1 The Effects of Temperature and Gas Mixture Composition on the Microstructure and Tribological Properties of the Plasma Nitrocarburized DIN 100 CR6 Steel**, *M Fontes*, Federal University of Sao Carlos, Brazil; *V Baggio-Scheid*, Sao Jose dos Campos, Brazil; *D Machado*, Tecumseh Products Company, Brazil; *L Casteletti*, University of Sao Paulo, Brazil; *Pedro Nascente*, Federal University of Sao Carlos, Brazil

Nitrocarburizing is considered one of the most important thermochemical treatments for surface modification of metallic materials, and involves the simultaneous diffusion of nitrogen and carbon onto the surface. Understanding and controlling the formation of the nitrocarburized layer have considerable industrial interest due to the improvements regarding wear, fatigue, and corrosion resistances. In this study, the DIN 100Cr6 steel was chosen due to its use as raw material in the manufacture of a mechanical component applied in hermetic compressors for refrigeration. The DIN 100Cr6 steel samples were treated by plasma nitrocarburizing for two hours, with two treatment temperature (550°C and 600°C) and four methane concentrations in the gas mixture composition (0%, 1.0%, 1.5%, and 2.0%) as variables. X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy dispersive spectroscopy (EDS) analyses, as well as wear resistance and micro-hardness tests, were used to characterize the modified samples. The results showed that the treatment temperature and atmosphere composition had considerable influence on the compound layer composition and morphology. The presence of carbon in the gas mixture contributed to the formation of the  $\epsilon$ -Fe<sub>2.3</sub>N phase, which has a HCP crystalline structure, and elevated temperatures caused an increase in the thickness of the compound layer, diffusion zone, and micro-porosity layer. The nitrided samples had a compound layer composed only by the  $\gamma'$ -Fe<sub>4</sub>N phase having a FCC structure. For the nitrocarburized samples, the compound layer was a mixture of  $\epsilon$ -Fe<sub>2.3</sub>N and  $\gamma'$ -Fe<sub>4</sub>N phases, with a columnar-like microstructure; the amount of each one of these phases was a function of the CH<sub>4</sub> percentage present in the treatment atmosphere. A micro-porosity layer was formed for all produced surface layers. A larger micro-porosity layer thickness was observed for samples nitrocarburized without CH<sub>4</sub>. Higher nitrogen concentrations in the atmosphere resulted in more pores in the compound layer.

8:20am **G2-2 Selected Aspects of Industrial Applications of Hydrogen Free DLC Coatings Deposited by CVAE**, *Joerg Vetter*, Oerlikon Balzers Coating Germany GmbH, Germany; *J Karner*, Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein; *J Becker*, *M Markus*, Oerlikon Balzers Coating Germany GmbH, Germany; *N Beganovic*, Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein; *E Billot*, Oerlikon Balzers Coating Germany GmbH, Germany; *H Rudigier*, Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein, Switzerland

The number of industrial applications of hydrogen free ta-C and a-C coatings continues to increase, primarily for tribological coatings to reduce wear and friction. Tetrahedrally-bonded hydrogen-free coatings (ta-C) provide the highest hardness, and are successfully applied in many cutting and forming applications, while various softer a-C coatings are also useful in various tribological applications. Recent research and industrial solutions for generating carbon-based coatings by CVAE (cathodic vacuum arc evaporation) are described. The performance of the coatings are influenced both by process parameters (coating architecture) and by topographical effects. Laboratory scale tribological investigations will be presented. Selected aspects of basic prerequisites for industrial applications are highlighted.

8:40am **G2-3 Erosion Resistant PVD Coatings for Gas Turbine Compressor Blades**, *Lin Shang*, *C Acikgoz*, Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein; *S Moser*, *G Szyndelman*, Oerlikon Metco AG, Switzerland; *O Jarry*, Oerlikon Balzers, Oerlikon Balzers Coating Germany GmbH, Germany; *M Arndt*, Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein

Although advancements in turbine materials have contributed to enhancing engine power ratings and efficiency levels, solid particle erosion (SPE) which occurs most prominently in the compressor section of aircraft engines during taking-off and landing still remains as a critical issue since the consequence removal of the turbine blade material degrades the aerodynamic performance, inducing reduced engine efficiency. Hence protecting turbine blades against erosion by applying PVD coatings can be a solution to maintain high level engine performance and lower maintenance costs.

In this work, TiAlN based coatings deposited by cathodic arc were applied on IN718, TiAl6V4 and stainless steel substrate materials. The influence of thermal exposure, coating thickness, hardness and residual stress on the SPE resistance of the coatings have been studied. SPE tests have been performed at 20° and 90° impact angles. Furthermore, the salt spray corrosion, SPE by water jet and cavitation resistance and fatigue behaviour of these coatings have been tested. The water jet erosion test was performed at different angles and also compared to HVOF sprayed WC-CoCr based coating. Compared to the uncoated substrate materials, the PVD coated substrates have been proven to have much higher erosion resistance.

9:00am **G2-4 Synthesis and Characterization of Ta-C, Hf-C and Ta-Hf-C Coatings Obtained by Cathodic Magnetron Sputtering in Reactive Conditions**, *Alexis de Monteynard*, Nogent International Center for CVD Innovation, LRC CEA-ICD LASMIS UMR6281, UTT, Antenne de Nogent, France; *A Billard*, Institut FEMTO-ST, CNRS, UTBM, Univ. Bourgogne Franche-Comté, Site de Montbéliard, France; *F Sanchette*, Nogent International Center for CVD Innovation, LRC CEA-ICD LASMIS UMR6281, UTT, Antenne de Nogent, France

Ultra-high temperature ceramics (UHTCs) have received a particular interest due to their high melting point leading to a well thermal protection for structures in extreme conditions [1]. The Ta-Hf-C system offers the possibility to create binary or ternary alloys having a melting point above 4000 K. Structural and thermal properties of bulk materials (TaC, HfC and Ta-Hf-C with a wide range composition) have already been investigated [2]. Influence of non-reactively magnetron-sputtering deposition parameters on Ta-C and Hf-C coatings properties has been studied, showing a strong impact of carbon content on structure, morphology, mechanical properties as well as thermal stability [3].

Ta-C, Hf-C and Ta-Hf-C thin films were deposited by cathodic magnetron sputtering of pure Ta and Hf targets in reactive condition (CH<sub>4</sub> being the reactive gas). Structural, morphological and mechanical properties have been studied. The influence of carbon content on coatings properties is discussed.

[1] W. G. Fahrenholtz, E. J. Wuchina, W. E. Lee, and Y. Zhou, Eds., Ultra-High Temperature Ceramics: Materials for Extreme Environment Applications. Hoboken, NJ: John Wiley & Sons, Inc, 2014.

[2] O. Cedillos-Barraza et al., "Investigating the highest melting temperature materials: A laser melting study of the TaC-HfC system," Sci. Rep., vol. 6, p. 37962, Dec. 2016.

[3] H. Lasfargues et al., "Non-reactively sputtered ultra-high temperature Hf-C and Ta-C coatings," Surf. Coat. Technol., vol. 309, pp. 436-444, Jan. 2017.

9:20am **G2-5 Thin and Thick Coatings and Applications in Aerospace Industry**, *Satish Dixit*, Plasma Technology Inc., USA **INVITED**

Coatings for wear, erosion, corrosion etc. have been implemented on over 500 different applications in aerospace industry. This includes, aircraft's used for commercial as well as military purposes along with critical components used in the Space exploration applications. In this talk I will be primarily focusing on thin and thick film coatings particularly applicable to functional aerospace components that are subjected to severe wear, corrosion, erosion etc. Thin films are critical where post processing is not an option and thick films are essential on more robust applications where endurance and strength is desired. I will be highlighting some of the applications developed in house as well as the applications prevalent predominantly within the industry at large.

# Tuesday Morning, April 24, 2018

10:00am **G2-7 HNT-Containing Ceramic PEO Coatings for Active Corrosion Protection of Magnesium Alloys**, *B Mingo, Yue Guo, A Matthews, A Yerokhin*, The University of Manchester, UK

The growing interest for magnesium in weight-sensitive applications has triggered the development of surface modifications techniques capable of improving its properties mainly, its corrosion resistance. Amongst them stand out Plasma Electrolytic Oxidation (PEO), which is a high voltage electrolytic-plasma surface treatment capable of obtaining highly stable ceramic coatings with excellent hardness, adhesion, corrosion and wear resistance. However, these coatings only provide passive protection, i.e. act only as a physical barrier between the metallic substrate and the aggressive environment.

The aim of this study is to develop a functional ceramic coating on a commercial magnesium alloy capable of interacting with its surrounding by responding selectively to specific triggers. For that, halloysite nanotubes (HNT) are incorporated into the coating, which can be loaded with different active agents such as corrosion inhibitors, lubricants or drugs.

Halloysite nanotubes are biocompatible natural clays composed by two layers of aluminosilicates arranged in a hollow tubular shape. These nanotubes can be loaded by mechanical (vacuum-induced capillarity) or chemical (ion-exchange) processes, so the release of their content can be triggered by different stimulus e.g. mechanical damage, time or pH variations. At neutral pH the inner part of HNT is positively charged, which means that it is able to host negatively charged agents attracted by electrostatic interactions, however when increasing pH the charge of the lumen of the nanotube changes, which forces the release of the incorporated agent. This is especially interesting for corrosion protection of magnesium alloys, where corrosion inhibitors can be released to the media when detecting electrochemical activity arisen from pH variations, remaining encapsulated while the coating is intact.

The main challenge faced in this work is to achieve the non-reactive incorporation of the loaded nanotubes to PEO coatings since the high temperatures and pressure reached during the coating synthesis might compromise the 3D integrity of the nanotubes. The obtained materials are evaluated in terms of characterization and corrosion resistance.

A positive outcome would not only increase the life-time of PEO coated components used in high performance applications, but also would expand their applicability to other fields, potentially to biomaterials, with the development of drug-loaded coatings used in orthopaedic implants.

## Surface Engineering - Applied Research and Industrial Applications

### Room Sunset - Session G6

#### Application-driven Cooperation Between industry and Research Institutions

**Moderators:** Tobias Brögelmann, Surface Engineering Institute - RWTH Aachen University, Joern Kohlscheen, Kennametal GmbH, S.P. Kumar Yalamanchili, Oerlikon Balzers, Oerlikon Surface Solutions AG

**1:50pm G6-2 Performance Evaluation of Precious Metal Coatings in Precision Glass Molding, Marcel Friedrichs, A Saksena, M Hans, RWTH Aachen University, Germany; O Dambon, Fraunhofer Institute for Production Technology IPT, Germany; J Schneider, F Klocke, RWTH Aachen University, Germany**

Precision Glass Molding (PGM) is a replicative technology for producing complex optical components with high surface quality and form accuracy. The efficiency of PGM process depends primarily on the lifetime of the high-precision molding tools made of cemented tungsten carbide. During each molding cycle, the molding tools have to withstand severe thermochemical and thermo-mechanical loads. Using protective coatings, the lifetime of the molding tools can be increased. The most versatile coatings for molding various glass types are based on precious metals due to reduced chemical interaction in contact with heated glass.

In this study, platinum-iridium (Pt-Ir) protective coatings were deposited on cemented tungsten carbide molding tools by physical vapor deposition (PVD) process. The investigated Pt-Ir coatings differed in their chemical composition. Based on predictions of the enthalpy of mixing, additional ternary Pt-Ir-X (X = Cu, Au) coatings were evaluated. To investigate the operational capability of the protective coatings, molding tests were carried out at an in-house built lifetime testing bench and at an industrial glass molding machine. Subsequent analyses of coated specimens were performed by white light interferometry and scanning electron microscopy (SEM). Depending on the chemical composition of the coating, different degradation phenomena as glass adhesion, interdiffusion and coating delamination were observed and discussed.

**2:10pm G6-3 Plasma-dependent Phase Formation of TiAlN Coatings, Anders Eriksson, Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein; M Hans, S Mráz, J Schneider, RWTH Aachen University, Germany; M Arndt, Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein**

The industrial success of TiAlN as wear-resistant and protective coatings is enabled by the well-known favourable age-hardening characteristics. However, the ability to control microstructure, mechanical and thermal properties of the coatings is also essential for application-specific coating design. In this contribution we study the deposition process of coatings deposited from  $Al_6Ti_{33}$  (at%) targets. By varying the arc source settings, the resulting coating properties have been optimized in industry for different cutting tool applications: either a single phase cubic solid solution or a mixture of cubic and hexagonal phase was obtained. Gaining in-depth understanding of the deposition processes leading to the different phase formation scenarios is a topic in our cooperation for application-inspired fundamental research within an industrial-scale deposition system. For the two arc source settings, we have characterized the plasma composition and ion energies in an Oerlikon Balzers INGENIA P3e™ deposition system by energy-resolved mass spectrometry. The phase formation was observed to depend significantly on the arc source configuration and hence plasma properties. Our findings illustrate that a collaboration between industry and academia enables knowledge-based design of protective coatings for specific applications.

**2:30pm G6-4 Reactive HiPIMS Deposition of Ti-Al-N: How to Adjust the Cubic to Wurtzite Transition, Helmut Riedl, L Zauner, P Ertelthaler, CDL-AOS at TU Wien, Austria; T Wojcik, TU Wien, Institute of Materials Science and Technology, Austria; H Bolvardi, Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein; S Kolozsvári, Plansee Composite Materials GmbH, Germany; P Mayrhofer, TU Wien, Institute of Materials Science and Technology, Austria**

High power impulse magnetron sputtering (HiPIMS) is often seen as one key-technology in the deposition of future hard and multifunctional coating materials. Through the introduction of high amplitude impulses at relatively low duty cycles, the amount of ionized species, either target near

gas atoms or sputtered target atoms, can be increased drastically. These highly dense plasmas have various consequences on the film growth and hence coating properties, as well as on the sputter behavior of the target material itself. Applying reactive gas mixtures such as  $N_2/Ar$  atmospheres, e.g. for the deposition of TiN coatings, lead to further complex effects within the plasmas (self-sputtering, gas recycling, or poisoning). However, several studies clearly highlighted the outstanding coating properties as well as metastable phases accessible, using HiPIMS compared to conventional DC magnetron sputtering, whereas a majority of these investigations concentrate on the plasma physics itself.

Therefore, we focused in this study on the reactive HiPIMS deposition of Ti-Al-N coatings using  $Ti_{1-x}Al_x$  compound targets (x = 0.50, 0.60, 0.66, 0.80) in mixed  $Ar/N_2$  atmospheres. The influence of the HiPIMS parameters such as frequency, pulse length, and power density, but also of the deposition parameters like partial pressure, deposition temperature, or total pressure was investigated methodically. The so obtained coating structures were analyzed with respect to phase stability, thermomechanical properties, and morphology applying nanoindentation, X-ray diffraction combined with electron imaging techniques (SEM and HR-TEM). In addition, to gain an in-depth understanding of the HiPIMS parameters on the used compound target materials a special in-situ target-temperature measurement system was utilized during all depositions (min. distance to sputtered target surface about 1 mm).

**2:50pm G6-5 AlTiN Coatings deposited by HIPIMS: A Study of Mechanical Properties, Tribological and Wear Performance during Machining of Superduplex Stainless Steel, J Paiva, Edinei Locks, Y Seid Ahmed, P Stolf, J Dosbaeva, McMaster University, Canada; C Bork, IFSul - Federal Institute Sul-rio-grandense, Brazil; G Fox-Rabinovich, S Veldhuis, McMaster University, Canada**

AlTiN PVD coatings represent a generation of coatings designed to work at high temperature applications. The aluminum in the film converts to aluminum oxide as the coating heats up, resulting in increased oxidation resistance. The use of this coating is related to the cutting tool applications where a lot of heat is generated. In this work, an advanced High Power Impulse Magnetron Sputtering (HIPIMS) technique was utilized to deposit dense AlTiN coatings on cemented carbide cutting inserts. The influence of HIPIMS process deposition was compared with AlTiN PVD Coating deposited by Arc method and evaluated in terms of microstructure, mechanical and tribological properties, by means of SEM /EDS, XRD structural characterization, nanoindentation testing, and Pin-on disc high temperature tribotesting. To relate the coatings properties with the wear performance, cutting tests were performed during turning of superduplex stainless steels at finishing operations. FEM modeling of the turning process was employed to determine the cutting temperatures, cutting forces and stresses at the cutting tool edge. The results obtained demonstrate that the tribological and wear performance of the AlTiN PVD coatings deposited by HIPIMS showed significantly improved wear behavior as compared to arc deposited AlTiN coating. This is because HIPIMS process allows to deposit very dense, defect free coatings, with low residual stresses and excellent surface finish that improves wear performance of the coated tool.

**3:10pm G6-6 FunMat-II – an Industry-Academia Competence Center for Research on Coating Materials for Advanced Applications, Lina Rogström, M Odén, I Abrikosov, G Greczynski, P Eklund, E Björk, Linköping University, IFM, Sweden**

FunMat-II (Functional Nanoscale Materials) is a second-generation competence center in material science, continuing the successful VINN Excellence Center FunMat inaugurated in 2007 (ended 2016). The center starts late 2017 and will continue for five years. FunMat-II is financed by the Swedish Agency for Innovation Systems (VINNOVA), Linköping University and our 12 industry/institute partners.

FunMat-II is focusing its efforts to three application areas for functional surfaces: cutting tools, fuel cells, and batteries. We obtain basic knowledge about materials behavior and the physics and chemistry of the synthesis processes, and design new materials with unique properties. We study all aspects using combinations of theory, modeling, experiments, and field tests. The information obtained is generic and can be applied to a wide range of applications, which makes FunMat-II a true competence center in functional surfaces optimized at the nanoscale.

This presentation gives an overview of the partnership and the way of working within the competence center, including application-inspired fundamental research, industry-oriented PhD education, continuing education, and intellectual property handling. Scientific highlights from

# Tuesday Afternoon, April 24, 2018

recent studies of materials for advanced application are given as examples, including high-temperature contacts [1], *in situ* characterization of hard coatings in turning operations, and experimental and theoretical studies of coatings with improved thermal stability for high-speed cutting [2-4].

[1] H. Fashandi et al., Nat. Mater. 16(8) (2017) 814.

[2] L. Rogström et al., J. Appl. Phys. 118 (2015) 035309.

[3] I. Schramm et al., Acta Mater. 119 (2016) 218

[4] F. Wang et al., Acta Mater. 127 (2017) 124.

**3:30pm G6-7 Oxygen Diffusion Pathways in High Temperature Oxidation Resistant Ti-Al-N/Mo-Si-B Multilayer Coatings**, *Elias Aschauer*, CDL-AOS at TU Wien, Austria; *P Felfer*, Friedrich-Alexander-Universität Erlangen-Nürnberg, Germany; *M Arndt*, Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein; *P Polcik*, Plansee Composite Materials GmbH, Germany; *H Riedl*, CDL-AOS at TU Wien, Austria; *P Mayrhofer*, Institute of Materials Science and Technology, TU Wien, Austria

In high temperature oxygen containing atmospheres, the oxidation resistance of a protective coating is mainly based on the ability of the participating metals to form adherent and continuous oxide scales separating the reactants from the oxidizing atmosphere. Corundum type  $Al_2O_3$  or  $Cr_2O_3$  represents such highly stable oxide structures, whereby the formation of Al-rich scales was crucial in the success story of  $Ti_{1-x}Al_xN$ . Nevertheless, oxide scales only constitutes semipermeable barriers slowing down scale growth, where the kinetic of the growing scale is determined by the fastest species - e.g. metal or oxygen ion outward and inward diffusion, respectively. With respect to the temperature ( $T < 0.6 T_m$ ), the dominant transport mechanism in growing scales is along fast-diffusion pathways such as dislocations, voids, or especially column boundaries rather through the bulk crystal lattice. To further enhance the oxidation resistance of e.g.  $Ti_{1-x}Al_xN$ , a fundamental understanding of the oxidation process and a distinct knowledge on the phase evolution and present diffusion pathways in the atomic scale range is highly desired.

Therefore, we used atom probe tomography (APT) to analyse the diffusion pathways in our high-temperature oxidation resistant Ti-Al-N/Mo-Si-B multilayer coatings. The repeated incorporation of very thin sputtered Mo-Si-B layers ( $\lambda \approx 25$  nm) in arc-evaporated Ti-Al-N ( $\lambda_i \approx 100$  nm) leads to an interrupted growth of the V-shaped Ti-Al-N columns, and hence to highly distinct areas in 3D chemical mapping. To overcome the difficulty of mass/charge peak overlap during APT investigations, the samples were annealed in  $O^{18}$  for 60 min at 900 °C. For optimized volume analysis, three tips parallel to the layered structure, next to the growing scale, were prepared by a standard FIB lift out technique – uniformly distributed from top to bottom in the unaffected coating region. These results were correlated with a detailed analysis on the phase evolution applying nano-beam X-ray diffraction as well as morphology by using high-resolution transmission electron microscopy in the as deposited and thermal treated state.

**3:50pm G6-8 Novel ta-C Coatings with Outstanding Tunable Properties Deposited by Industrially Scaled PLD**, *Martin Hess*, Fritz Stepper GmbH & Co. KG, Germany; *S Weißmantel*, *R Bertram*, Hochschule Mittweida, Germany

ta-C deposited by PVD is successfully introduced for quite some time for wear protection applications of engine components (piston rings, injection parts, etc.). Due to their geometrically relatively simple functional areas, batching as well as a posttreatment of coated surfaces is common compared to more complex shaped parts. Due to its prominent mechanical properties ta-C is actually increasingly introduced to commercial PVD-systems. However, limitations are high intrinsic stresses, roughness and also tribochemistry. As a consequence ta-C is still rarely found on industrial tools for stamping and chip removing processes.

Acting in the spirit of our company founder ("If you want to improve something, you have two options: Optimizing existing procedures or rethinking the whole process") we decided in early 2016 not to add another PVD-system to our equipment park (CARC, PECVD, HIPIMS). We thought rather about a surface enhancement technology which would be dedicated and designed towards our internal requirements to maximize quality of small, one-digit ccm volume, parts in batches of only some 10 to a few 100 pieces per day.

Our primary motivation was that in one of our high performance modular stamping tools up to 5000 moving components are facing stroke rates exceeding 2000 strokes per minute. In this context our boosting ambition is the creation of maintenance free tools up to production lots of 100 million electrical contact parts and more. However even latest PVD-coatings on

carbide substrates of highest quality are actually limited to some million strokes before showing critical wear leading to expensive tool downtime. As abrasion was isolated as the dominant life-time limitation our obvious conclusion was to seek for significantly higher abrasion resistance than our latest 35-40 GPa nanolamellar or nanocomposite PVD coatings. At the end ta-C was assessed as the most promising evolution.

Preceding benchmark tests regarding the ta-C coating process options isolated the so far for wear protection applications relatively unknown or rather unusual PLD as a promising process, due to some unique inherent features: smooth surfaces, homogeneous erosion of the target and last but not least: precise control of mechanical coating properties such as Y and H as well as tunable intrinsic stresses by laser energy. As a result smooth and well adherent coatings of up to 6  $\mu m$  thickness were deposited on steel and carbide substrates. The purpose of the present contribution is to give an introduction to this novel technology and its first successful applications.

**4:10pm G6-9 Application-driven Cooperation Between Industry and Research Institutions: Success Factors, Obstacles and Success Stories**, *Oliver Lemmer*, *W Koelker*, CemeCon AG, Germany **INVITED**

Development of new products and new technologies should be triggered by market needs. Depending on the respective subjects and the economic constraints, the market expects those needs to be met either in short term or in mid/long term. Subsequently two major challenges have to be mastered:

1. The different time line expectations have to be addressed by different styles of cooperation and by selectively chosen partners, in order to succeed in creating new products and technologies.

1. The "chicken and egg" problem: new products often need new technologies to produce them.

Recently, two new technologies have gained prominent importance for advanced coatings for demanding applications, e.g. in aerospace industry: HiPIMS technology and CVD diamond technology. In this context, the different roles of partners in R&D-networks will be described, and the different objectives and tasks with respect to the different time horizons will be presented. An outlook will be given on the potential of new products created by the combination of these two high-end technologies.

## Surface Engineering - Applied Research and Industrial Applications

### Room Sunset - Session G1

#### Advances in Industrial PVD, CVD, and PCVD Processes and Equipment

**Moderators:** Emmanuelle Göthelid, Sandvik Machining Solutions, Ladislav Bardos, Uppsala University, Sweden

##### 8:00am G1-1 Enhanced PVD Process Control by Online Substrate Temperature Measurement, *K Bobzin, T Brögelmann, Nathan Kruppe*, RWTH Aachen University, Germany

In physical vapor deposition (PVD) processes pressure and temperature are of outstanding importance for process control and coating performance. The pressure measurement and its integration into the process control are state of the art in industrial coating units. In contrast, the precise determination of the substrate temperature  $T_{\text{Substrate}}$  presents an unequally greater challenge. In PVD processes the temperature control has a great importance in two respects. On the one hand, the morphology of the coating as well as the adhesion between the coating and substrate directly depend on  $T_{\text{Substrate}}$ . On the other hand, the process temperature is limited by the maximum permissible temperature  $T_{\text{Max}}$  as for example of heat-treated steel. Exceeding can easily lead to a critical loss of hardness and size accuracy of the substrate and production reject. The online measurement of  $T_{\text{Substrate}}$  using currently applicable systems such as drag pointer or pyrometer is either extremely complicated or simply not possible. This is particularly the case for large-volume industrial coating units with a rotating substrate table. Therefore, in coating processes, the influence of the different process parameters on  $T_{\text{Substrate}}$  is usually not quantified. Hence,  $T_{\text{Substrate}}$  is often several tens centigrades below  $T_{\text{Max}}$ . Within the scope of this paper, a temperature sensor developed at the Surface Engineering Institute (IOT) of the RWTH Aachen University is used in an industrial coating unit with rotating substrate table. With this sensor, the online temperature measurement of rotating substrates is possible throughout the entire coating process, from the heating to the etching over the coating to the cooling process phase. The measuring accuracy is  $\Delta T = \pm 1$  °C. In all four process phases, the influence of the heating power on  $T_{\text{Substrate}}$  was analyzed and mathematically described. The results were used to improve the temperature management of an industrial coating process with regard to  $T_{\text{Max}}$ . Here the temperature difference between  $T_{\text{Max}}$  of the heat-treated steel and the realized  $T_{\text{Substrate}}$  was reduced to a minimum over the entire process time. In addition, the influence of the cathode power and cathode number for direct current (dcMS) and high power pulsed magnetron sputtering (HPPMS) processes as well as for hybrid processes dcMS/HPPMS on  $T_{\text{Substrate}}$  was quantified. For a hybrid six-cathode process, it was shown that an increase in the cathode power by 100 % results in an increase of  $T_{\text{Substrate}}$  by approx. 17 %. Investigations on the compound properties show a considerably improved adhesion between coating and substrate.

##### 8:20am G1-2 A Compact, Symmetrical and Efficient Filtered Cathodic Arc Source that uses Permanent Magnets, *Paul Sathrum*, Fluxion Inc., USA

A compact and symmetrical filtered cathodic arc source that uses no magnet coils is presented. The Radial Arc is described and compared to other filtered cathodic arc sources, including how permanent magnets are used instead of coils and how geometric factors promote efficiency. Compared to unfiltered cathodic arc, the Radial Arc is shown to retain many of its useful popular features such as simplicity and ease of maintenance and operation. Deposition rate profiles for metal and tetrahedral amorphous carbon (ta-C) films are given and show rates comparable to unfiltered arc.

##### 8:40am G1-3 HiPIMS Meets Diamond, *T Leyendecker, O Lemmer, W Kölker, Christoph Schiffers*, CemeCon AG, Germany

This paper will introduce a visionary new class of coating materials with revolutionary properties. It creates added value by merging diamond – the hardest of all materials – with HiPIMS – smooth and dense thin films.

The exceptionally hard diamond layer provides the perfect foundation to the HiPIMS film. Diamond has an outstanding thermal conductivity and spreads the extreme heat that is generated in the cutting zone. Both effects, the unrivalled hardness and the heat spreading properties of diamond, avoid the typical egg-shell effect. The generally lower heat input

into the carbide substrate and the avoidance of local overheating is an enormous plus when it comes to cutting of heat resistant superalloys.

HiPIMS is a vital contribution to this new material: HiPIMS films are dense and have thereby a high oxidation resistance. Quite a portion of the heat load during machining is transferred into the chips. The other beneficial effect of the HiPIMS part of the new material is its protection of the diamond against dissolution at high temperature in an oxygen atmosphere. The smooth, droplet-free surface of HiPIMS coatings reduces friction, heat generation and optimizes the running in process of the cutting tool.

Case studies like the machining of casted CrCo for medical implants and the milling of stacks with extra thick titanium layers show that the radically new materials concept HiPIMS meets Diamond is the answer to new business for cutting tools after the combustion engine.

##### 9:00am G1-4 Functional DLC by HiPIMS and Pulsed DC-magnetron Sputtering in an Industrial Coating System, *I Fernandez Martinez, A Wennberg*, Nano4energy, Spain; *F Papa*, Gencoa, Ltd, USA, Spain; *J Santiago*, Nano4energy, Spain; *N Dams*, PVT GmbH, Germany; *Herbert Gabriel*, PVT Plasma und Vakuum technik GmbH, Germany

The demand for high-performance coatings for special tooling and component applications is increasing rapidly. The most prominent example for such coatings are Diamond-like carbon (DLC) coatings, due to their tribological, wear resistant and corrosion resistant properties. Due to the growing importance of such coatings in many industrial fields, the market for industrial coating systems is growing very fast.

In order to fulfil the future's demand of such coatings, efficient and highly productive coating systems are required. The consortium's approach is an advanced industrial vacuum coating system with a plasma volume of 350 × 650 mm ( $\varnothing \times H$ ) equipped with four large area magnetron sputtering-sources and the new hiPlus - positive pulsed technology from Nano4Energy. The magnetrons, manufactured by Gencoa Ltd, incorporate the VTR technology (Variable Magnetic Field) that allows to vary the balanced to unbalanced degree of the magnetron source during deposition, and thus, the ion bombardment of the growing film.

The plasma can be excited in both HiPIMS and DC-Pulsed mode, that in combination with an optimum adjustment of the magnetic field design, smooth coatings with high hardness and ideal adhesion can be produced efficiently. Detailed process description will be given.

The technology is very much up-scalable.

A wide variety of DLC coatings can be deposited in the system with hardness values up to HV 4000. This includes hydrogen-free amorphous carbon (a-C), hydrogenated amorphous carbon (a-C:H), tetrahedral amorphous carbon (ta-C) or Metal-doped DLC (such as Cr-doped DLC). Detailed information on coating properties will be given.

As a benefit, the design of the system allows the deposition of extremely smooth hard nitrides and carbo-nitrides, such as all standard multi-layered, nano-structured TiN, TiCN, AlTiN, AlCrN and Si-doped coatings.

For industrial production purposes the system runs in a fully automatic mode. For R&D - applications the system can be operated in a complete manual mode.

##### 9:20am G1-5 Microwave Assisted PVD and PECVD Systems for Carbon-Based Nano Composites, *Sven Ulrich, C Poltorak, M Rinke, H Leiste, M Stüber*, Karlsruhe Institute of Technology (KIT), Institute for Applied Materials (IAM), Germany

**INVITED**

HiPIMS differs significantly from conventional d.c. magnetron sputtering, and together they cover a wide range of scenarios of deposition kinetics that are often critical for the characteristics of resulting coatings. In d.c. magnetron sputtering, particles sputtered from the target are mostly electrically neutral with low kinetic energies of only a few eV. In HiPIMS deposition, however, the particles sputtered from the target crossing the highly densified plasma are largely ionized and, therefore, their kinetic energy can be purposefully adjusted before arriving at the deposition surface via an appropriate substrate bias. Additionally a novel microwave plasma source designed for large-area industrial plasma surface processing and film deposition creates high density plasmas at low pressures even far from the source. A hybrid deposition method (HiPIMS, d.c. magnetron sputtering and microwave plasma source deposition) is developed for advanced carbon-based nanocomposites in the system Ti-Zr-C-N. The coating properties and microstructures were characterized and compared. The results will be discussed in this study and related to the strongly different deposition kinetics. The coatings were deposited from a segmented metallic Ti/Zr target in reactive mode with different Ar-N<sub>2</sub>-CH<sub>4</sub>

# Wednesday Morning, April 25, 2018

gas mixtures. Effects of thermal treatment were also examined on the deposited coatings. The chemical composition of coatings was analyzed by electron microprobe, the morphology by scanning electron microscopy (SEM), and the microstructure by X-ray diffraction (XRD). The coating hardness as well as reduced elastic modulus was characterized by nano-indentation.

**10:00am G1-7 Correlation Between Plasma Nitriding of Several Steels and Active Nitrogen Concentration Correlated through Optical Emission Spectroscopy and Atomic Nitrogen Partial Pressure**, *F Papa*, Gencoa, Spain; *Joaquin Oseguera*, TRAMES S.A. de C.V., Mexico

Plasma nitriding of steels has been a very important industrial solution to attend solicitations that are produced on many tribo-mechanical applications. Nitrogen diffusion into the steel can be correlated to specific spectral lines obtained from optical emission spectroscopy in the plasma, active nitrogen can be identified through this technique. The correlation between parameters to produce nitrogen diffusion in the steel makes relevant to have a reference of the active species in the process. Analyses of spectral lines observed from the plasma indicate zones where the active nitrogen can produce diffusion into the steel. Through this work, several steels were treated in weak ionized plasma; active nitrogen identified from OES was correlated with atomic nitrogen partial pressures measured from remote emission spectroscopy, through the analysis of information a procedure to identify plasma parameters to produce nitrogen diffusion in steels is presented.

**10:20am G1-8 CVD Technology & Machinery – Tribological Applications and High Temperature Potential**, *Hristo Strakov*, *V Papageorgiou*, *M Auger*, IHI Ionbond AG, Switzerland

Hard material coatings deposited by thermal CVD have maintained their outstanding position for decades in the field of many tribological and high temperature applications. This success can be explained by the excellent mechanical properties at high temperatures and the thermochemical stability of the coatings. Through continuous further development of the materials and the coating machinery, today's coating products are adapted and optimized for the constantly growing demands on the composition and structural properties. Moreover, new CVD technologies are also being developed that address friction and wear improvements at elevated temperatures. Examples of these developments include high Aluminum content TiAlN. Optimized coatings can be produced through specific multilayers adaptations, coating material combinations, together with suitable substrate materials. Specific examples illustrating the influence of coating parameters and base material condition upon performance as well as the necessary equipment improvements are presented.

**10:40am G1-9 Vacuum Barrel Coating: An Opportunity to Performance Increase for Various Small Parts**, *Heidrun Klostermann*, *B Kraetzschmar*, *F Fietzke*, Fraunhofer FEP, Germany

Vacuum coating has found its way into many sectors of mechanical engineering due to the benefits of suitably coated surfaces in terms of wear resistance, corrosion protection and other value-added properties. Essential criteria for the implementation of a vacuum coating step in production are the technical, the economic and the ecological viability. The latter is a pro of vacuum coating processes, the other two challenge engineers as well as entrepreneurs. In the coating of three-dimensional parts, the handling of these represents a considerable fraction of the whole effort for coating. Handling individual parts, often in several process steps, is still mainly a manual task. It is not an option for a large quantity of small parts, which have to be handled as bulk goods.

Vacuum barrel coating seems to be an appropriate technique for the finishing of bulk goods that have to withstand severe load conditions in service. However, coating and process development for a variety of very different applications is a challenge for researchers and engineers. Fraunhofer FEP has pursued the objective of vacuum bulk coating for some years. In this talk, we want to share our experience regarding plasma activated high rate evaporation and magnetron sputtering in the barrel coating device ALMA 1000. Evaporated aluminum based coatings, sputtered coatings, as well as multilayers will be evaluated in the context of their respective field of application and an outlook will be given to further development options.

**11:00am G1-10 Scaling Up Graphene-like Carbon Film: Insights into the Deposition Process in a Roll-to-roll rf Plasma CVD System**, *Majed Alrefae*, *A Kumar*, *D Zemlyanov*, Purdue University, USA; *T Fisher*, UCLA, USA

Graphene and graphene-like-carbon films have found broad applications ranging from corrosion protection of metals to transparent conducting

electrodes. Here, we study a high-throughput, roll-to-roll plasma chemical vapor deposition (CVD) of graphene-like-carbon film on Cu foil, particularly the related effects of heat transfer and web speed on film quality in combination with optical emission spectroscopy (OES) to study plasma composition during growth. Graphene-like carbon film is deposited on a moving copper foil using a custom-built roll-to-roll radio-frequency plasma CVD system. The carbon film is deposited on both sides of the substrate at a high speed of 300 mm/min. We find an asymmetry in the quality of the film on the two sides of Cu foil attributed to asymmetry in the plasma's sheath. Results from OES provide insights into the distribution of chemical species between the electrodes. Emission intensities from major plasma species (i.e., CH, C<sub>2</sub>, H and H<sub>2</sub>) show no dependence on web speed. However, the temperature of the Cu foil, estimated from its blackbody emission using OES, decreases with increased web speed. The Cu foil temperature is near 1000 K for plasma power of 1350 W. We also find that high web speed is necessary with high-energy plasma input to achieve an optimal dwell time and minimal defects in graphene-like carbon film. The results provide insights into roll-to-roll growth of graphene-like carbon film on Cu foil using heat transfer principles and establish the feasibility of the method for a large-scale production.

**11:20am G1-11 TAOS Based Cu/TiW/IGZO/Al<sub>2</sub>O<sub>3</sub>/Pt Bilayer CBRAM for Low-power Display Technology**, *Kai-Jhih Gan*, *P Liu*, *W Chang*, *D Ruan*, *T Chien*, *Y Chiu*, *S Sze*, National Chiao Tung University, Taiwan

Herein we report a Cu/TiW/IGZO/Al<sub>2</sub>O<sub>3</sub>/Pt bilayer CBRAM stack using amorphous InGaZnO, a TAOS material, as the resistive switching layer. The addition of a thin metal-oxide layer (5 nm-thick Al<sub>2</sub>O<sub>3</sub>) in the bottom of the IGZO memory stack significantly increases the R<sub>OFF</sub> and the memory window. The a-IGZO bilayer CBRAM shows the excellent memory performances, such as low operation current (down to 10μA), high on/off resistance ratio (more than 10<sup>3</sup>), high switching endurance (up to 10<sup>3</sup> cycles) and the capability of multi-level tuning have been demonstrated in our memory device. Meanwhile, high thermal stability was also achieved (two decades of window margin are constantly maintained beyond 10<sup>4</sup> s at 85 °C). This result has given a great potential for the TAOS based material utilizing in CBRAM stacks and integrating into the display circuit for future memory in pixel application.

## Surface Engineering - Applied Research and Industrial Applications

### Room Sunset - Session G5

#### Hybrid Coatings and Hybrid System Processes

**Moderators:** Hana Barankova, Uppsala University, Sweden, Sang-Yul Lee, Korea Aerospace University

**1:50pm G5-2 Propagation of Electric Field Waves in a DC Magnetron Plasma, Rachel Broughton, S Kirkpatrick, Rose-Hulman Institute of Technology, USA**

Arcing is an issue that extends throughout the field of plasmas that can cause expensive damage to industrial equipment. In magnetron plasmas, arcing is usually avoided by employing a preventative method, such as pulsed DC sputtering. This work superimposes a small AC signal onto the high voltage supplied to the DC cathode to observe the effects within a magnetron plasma. A Langmuir probe was inserted into an argon DC plasma formed by a two-inch magnetron gun. The voltage from the probe was read on an oscilloscope not only in the time domain to obtain the appropriate IV-curve, but also in the frequency domain to observe if the driven frequencies were able to propagate through the plasma. An 8 mm by 8 mm square probe was employed both parallel and perpendicular to the surface of the cathode. Variations depending on orientation were noted in the measured plasma temperature and density where typical density values of the observed plasmas were on the order of  $10^{10} \text{ cm}^{-3}$ . The applied AC signal was detected in the time domain measurements of the Langmuir probes only from probes oriented perpendicular to the surface of the cathode.

**2:10pm G5-3 From Surface to Coating - Tools for Surface Engineering, Frank Papa, Gencoa Ltd, USA, Spain; V Bellido-Gonzalez, Gencoa Ltd, UK; I Fernandez Martinez, Nano4energy SLNE, Spain; F Meyer, H Li, D Monaghan, T Sgrilli, Gencoa Ltd, UK**

#### INVITED

Surface engineering has been an area of interest for many decades. Initially, hybrid processes such as nitriding, anodizing and plating were applied to improve the surface properties of engineering materials such as steels and aluminum. Today, we are faced with many surface engineering challenges as the number of advanced materials increases and the "functionality" requirements of surfaces become more demanding. From metals to plastics to ceramics, the types of surface treatments and coatings are varied. For surface preparation, the energy and type of ion needed (etching/functionalization/cleaning) will vary with the application. Likewise, the properties of coatings and surface coating interfaces depend strongly on the type of impinging particles and their energies and reactivity. Several tools are available for "hybrid" processes where the energy and type of ions reaching a surface need to be controlled. Ion sources are typically used for pre-treatment of substrates, but sputtering magnetrons themselves can also be turned into sources of ions. Conversely, both can be used for depositing coatings. Advanced tools for surface engineering in today's world involve an understanding of how sources can be driven (power supplies) and how combinations of technologies, both active (anode layer ion sources) and passive (anodes), allow us to change interfaces and coating properties. In this talk, we'll explore such applications in the areas of glass coating, hard coatings and on polymers. We'll discuss the combined use of secondary sources to boost and to decrease plasma density as well as the effects of adding positive pulses to traditional HIPIMS processes. With such positive pulses, the ion flux and energy can be controlled to the growing film after each negative pulse (deposition). The successful implementation of hybrid processes for coatings such as AlCrN, ITO, DLC and functionalized Ag will be presented.

**2:50pm G5-5 Nb – Doped TiO<sub>2</sub> Deposited by Hybrid HIPIMS – CVD Process, Justyna Kulczyk-Malecka, Manchester Metropolitan University, UK; D Donaghy, University of Liverpool, UK; B Delfour-Peyrethon, Manchester Metropolitan University, UK; P Chalker, J Bradley, University of Liverpool, UK; P Kelly, Manchester Metropolitan University, UK**

Novel methods for the deposition of thin functional coatings, such as hybrid CVD – PVD technologies have the potential to become an important means of overcoming the limitations of current processes, such as low deposition rates, associated with some sputtering processes or limited material/precursor choices, associated with CVD processes.

In this work we are combining PECVD with a magnetron sputtering source driven by a HIPIMS power supply to assist the deposition of functional

oxides for TCO applications. Although, other PVD – CVD hybrid systems have been developed, each process requires its own power supply. However, combining the processes in this way means that only one power supply is required. Thus, niobium – doped titania coatings were deposited on glass and Si wafer substrates by this hybrid HIPIMS – CVD technique. The TiO<sub>2</sub> coatings were deposited by CVD from a TTIP precursor via the vapour drawn method. The HIPIMS process provided not only the source of Nb metal dopant to the functional films, but sustained the low temperature CVD process by the means of a highly energetic plasma. Furthermore, since HIPIMS deposition rates are very sensitive to magnetic field strength and the degree of unbalance, by using a magnetron with variable magnetic field strength, it was possible to adjust the dopant content of the film without adjusting the power applied to the magnetron target.

The effect of processing parameters (pulse frequency, peak powers, precursor flow rates, operating pressure, etc.) on generating a stable HIPIMS plasma across the process envelope has been studied in this work. The composition, microstructure and electrical properties of the deposited coatings have been investigated, in respect to variable process parameters, such as substrate temperature and operating pressure.

**3:10pm G5-6 Potential of Sequent and Simultaneous PVD PeCVD Hybrid Technology Combination. Investigations Aside Well-known Technologies in Duplex DLC and Co-deposition by Simultaneous Arc, Sputtering Evaporation, Pierre Collignon, R Scheibe, PD2i Europe GmbH, Germany**

As Hybrid technology, the combinations of different PVD and PeCVD technologies like WC/C or Cr-DLC are state of the art technologies that are well established and industrialized but there are more possible idle technological combinations. Research was concentrated on two specific technology combinations:

1) Further investigations in DUPLEX DLC, an upstream NITRATION to increase surface hardness and increased resistance against plastic deformation with a subsequent tribological DLC coating reducing the coefficient of friction in one batch (INSITU). Goal of the study is discovering the properties (adhesion, friction coeff, corrosion) but also the potential to what extent existing sequent processes can be substituted and further, the feasibility of substituting used material / surface treatment combinations.

2) Co-deposition a simultaneous ARC and sputtering evaporation to combine the advantage of high adhesive hard coatings with embedded lubricant / tribological nanoparticles. Today's weakness in multi layered lubricant thin films are the limited working temperature of 300 °C and the fast elimination of the weak functional tribological layers by wear. Target of the test series is to figure out if lifetime can be increased significantly by embedding lubricant particles into the hard coating by simultaneous evaporation of a different hard coatings (e.g. AlTiN) via ARC and materials with low friction coefficient (e.g. WC/C) via sputtering evaporation.

**3:30pm G5-7 TiN Deposition using the Magnetized Hollow Cathode Activated Magnetron, H Barankova, Ladislav Bardos, Uppsala University, Sweden**

A new type of the magnetron, Magnetized Hollow Cathode Activated Magnetron with the target coupled with the hollow cathode magnetized by the magnetic field of the magnetron was tested in the reactive process of TiN deposition. Increased deposition rate compared to the Ti metal deposition rate was confirmed. The depositions as well as optical measurements were performed at several pressures in the reactor. The results of the TiN reactive deposition are presented and discussed, including the TiN deposition in pure nitrogen.

**3:50pm G5-8 Structural and Tribological Properties of Mixed Iron-titanium Borides Produced with Cathodic Arc Assisted Alloying and Electrochemical Boriding, Erkan Kacar, C Yelkarasi, S Timur, M Urgen, Istanbul Technical University, Turkey**

Borides of transition metals are very promising materials for a wide range of applications due to their high hardness, excellent wear, high temperature oxidation resistance and high thermal/electrical conductivity. Most widely used borides are iron and titanium borides. Iron borides are produced with diffusion based processes applied to iron and steel alloys. On the other hand titanium borides are produced either by boriding titanium or as coatings on different substrates using PVD or CVD methods. In this study we aimed to produce complex borides composed of Fe and Ti borides using hybrid process. For achieving this aim, low carbon steel substrates are alloyed with titanium using cathodic arc assisted process to produce iron-titanium alloy on the surface. This alloy is then borided using electrochemical boriding method. Produced mixed boride is characterized

# Wednesday Afternoon, April 25, 2018

with respect to its structure, morphology and mechanical properties using XRD, SEM, FIB and ultra microhardness measurements. Tribological properties of the layers are determined using reciprocating wear tests against alumina balls. Results of the investigation revealed that it is possible to produce ultra hard surface layers (hardness above 40 GPa) by this hybrid method with excellent wear resistance.

# Thursday Afternoon Poster Sessions, April 26, 2018

## Surface Engineering - Applied Research and Industrial Applications

### Room Grand Hall - Session GP

#### Symposium G Poster Session

**GP-2 Laser-clad Induced Reaction Synthesis of TiC/WC Reinforced Co-based Composite Coatings on Copper Alloy, Hua Yan, P Zhang, Z Yu, Shanghai University of Engineering Science, China**

Co-based composite coatings reinforced by nickel coated WC (Ni/WC) and in-situ synthesized TiC particles has been fabricated from precursor mixtures of HG-CoO1(Co-based alloy), Ni/WC, graphite and pure titanium powders by laser cladding on Cr-Zr-Cu alloy substrate. The microstructure, phase and wear properties were investigated by means of optical microscopy (OM), X-ray diffraction (XRD) and scanning electron microscopy (SEM), as well as dry sliding wear test. Results show that reinforcements dispersed uniformly in the Co-based matrix. TiC showed the morphology of dendritic and particle. During laser-clad processing, the laser heating effect caused inter-diffusion action between the precursor mixtures and generated Ti<sub>3</sub>Cy and some Ti element diffused into Ni/WC particles formation of TiWC<sub>2</sub> alloyed layer. The laser-clad TiC/WC reinforced Co-based composite coatings exhibited higher microhardness and better wear resistance than copper alloy. The highest microhardness was up to 1007 HV0.2 which was improved 8 times comparing to the Cr-Zr-Cu substrate. The friction coefficients of the laser-clad composite coatings were reduced significantly to about 0.15 and relatively smooth wear surface could be observed.

**GP-5 The Study of Mechanical Strength on the Injection Molding Parameters of PMMA/TG Composite Bipolar Plates, Ai-Huei Chiou, National Formosa University, Taiwan**

Recently, green energy technology is developing in the world and fuel cell is one potential energy resource of renewable energy technologies. Now fuel cell is facing the challenges of popularization and miniaturization. The bipolar plate is a key component in this device, so to develop highly conductive, lightweight, and low cost bipolar plate for fuel cell is important goal. Besides, the design of fuel cell assembly plays an important role in the performance. Therefore, the primary goal of this study is improving process, material selection and mechanical properties of bipolar plate. To make a breakthrough the bottleneck of science and technology in fuel cell and create more economic value in the future.

Therefore, weight and cost of bipolar plates were important research project for improvement. In this study, light-weight composite bipolar plates were prepared by injection molding. The thickness of the lightweight composite bipolar plates were thinner in this study. After the injection molding bipolar plate may have poor strength caused affection [to fuel cell assembly and battery efficiency. In order to make it have good strength, take the tea graphites (TG) as reinforcing materials that were made of recover tea leaves and mixed with Polymethylmethacrylate. The experimental parameters were planned by Taguchi method to improve strength and warping quality.

The experimental results showed that the increase of TG content decrease the PMMA flow and transmittance, but the bending strength and heat resistance increased in PMMA / TG composites. The single quality optimization of bending strength of the PMMA / TG composite bipolar plate was about 35.64 ± 1.9 Mpa, which was improved about 7.6% higher than the maximum bending strength of L<sub>8</sub> (33.13 ± 1.4 Mpa) in the orthogonal table. Results showed that the composite of interest qualified under the Department of Energy (DOE) requirements for weight and flexural strength for fuel cell bipolar plates. The study concludes that the process of PMMA/TG bipolar plates by injection mold may be used in fuel cell applications.

**GP-6 Real-time Analysis of Neutral Species from Atmospheric Plasma, Peter Hatton, A Rees, C Greenwood, S Bort, Hiden Analytical Ltd, UK**

Electrical plasmas at atmospheric pressures find increasing application in materials processing and other fields. Generation of the plasmas is not necessarily more difficult than at low pressures, but the plasmas can involve a wider range of particle species, produced through an increase in interactions at the elevated pressures. Identification of the active species assists the characterisation of the plasmas and the evaluation of equilibrium pathways involved, thus aiding the optimisation of particular systems. The work reported here is for two simple RF atmospheric plasmas

studied using a Hiden HPR20 EPIC quadrupole mass spectrometer. The measurements were made in real-time using the instrument's capillary inlet system to couple to the reactor. Test data for a mixture of helium and carbon dioxide clearly show the plasma dissociation of the carbon dioxide. The reaction was monitored either through observing the intensities of CO<sup>+</sup> and CO<sub>2</sub><sup>+</sup> ions produced in the source of the HPR20 EPIC from the sampled gas or the intensity of the negative oxygen ions produced by selecting the electron energies in the source to be those corresponding to the peaks in the dissociative electron attachment for the two gases.

Data from tests using a methane/helium plasma show clearly the wide range of high mass products produced by the plasma, and their rapid response to changes in plasma conditions. Negative ions such as H<sup>-</sup> and C<sub>2</sub>H<sub>2</sub><sup>-</sup> were monitored as well as high -mass positive ions. The data illustrate some of the potential of instruments such as the HPR20 for the study of high pressure plasmas, for example in the production of hydrophobic surfaces.

**GP-8 Correlation of HPPMS Plasma and Coating Properties using Artificial Neural Networks, K Bobzin, T Brögelmann, N Kruppe, Martin Engels, Surface Engineering Institute - RWTH Aachen University, Germany**

The development of industrial coating processes for tool coatings by means of physical vapor deposition (PVD) is usually extremely complex. This is caused by the large number of necessary coating batches and associated coating analyses until suitable process parameters are found. Artificial neural networks (ANN) are basically capable of describing complex relationships between various characteristic process values. Hence, within the scope of this paper the capability of describing complex correlations was tested on the example of a reactive high power pulsed magnetron sputtering (HPPMS) (Cr,Al)ON process. Selected process parameters pulse frequency and process gas composition were chosen, since they exhibit strongly non-linear cause-effect relationships. The ANN was used in order to correlate selective results from efficient substrate-oriented plasma diagnostics and coating analyses. Regarding the plasma properties the Al/Cr ratio and the metal-to-gas ion flux ratio were considered. With respect to the coating properties the Al/Cr ratio and the universal hardness were examined. From the correlation of these results, conclusions on the process parameters for desired coating properties were deduced and successfully proven for the investigated HPPMS (Cr,Al)ON process. Hence, the ANN exhibits a great potential to supplement the fundamental understanding of PVD processes in order to contribute to a simplification of the development of industrial coating processes.

**GP-9 Linking Erosion and Sputter Performance of a Rotatable Target to Microstructure and Properties of Mo Thin Films, A Hofer-Roblyek, K Pichler, Montanuniversität Leoben, Austria; C Linke, Plansee SE, Austria; R Franz, Montanuniversität Leoben, Austria; J Winkler, Plansee SE, Austria; Christian Mitterer, Montanuniversität Leoben, Austria**

The use of molybdenum in large area thin film deposition includes back contact layers for thin film solar cells as well as diffusion barriers and source/drain electrodes in microelectronics and relies on its excellent thermal stability and chemical inertness as well as low electrical resistivity. A constant high quality of sputter deposited thin films during the entire target lifetime is of vital importance for these applications. Thus, this study addresses the sputter performance, i.e. changes of current, voltage and arc rate, recorded during erosion of a rotatable Mo target as well as the quality of thin films deposited at different erosion stages. The enhanced target erosion and the thus reduced target wall thickness cause an increase of the magnetic field strength in front of the target and yield a slightly reduced voltage and increased current. Increased arc rates could be related to venting the vacuum chamber during interruptions in target erosion which were needed for thin film depositions. Both, microstructure and electrical resistivity of the films deposited are widely unaffected by the progressing target erosion. In contrast, the different substrate carrier oscillation modes determine film topography, stress and electrical resistivity. The end of target life is determined by the pronounced sputter grooves formed at both ends of the rotatable target due to the shape of the permanent magnetic field at the turnarounds rather than changes in the quality of the films deposited.

**GP-10 Surface Profile Analysis as an Investigative Tool for Electrolytic Plasma Polishing, Nicolas Laugel, A Matthews, A Yerokhin, University of Manchester, UK**

Electrolytic Plasma Polishing (EPPo) is a finishing method for the smoothing of conductive workpieces. Based on the electrodisolution of metals at electrical potentials lying in the hundreds of volts range, it presents the advantages of electropolishing. The absence of a tool, for example, means

# Thursday Afternoon Poster Sessions, April 26, 2018

independence from macroscopic surface geometry as well as the avoidance of tool wear. Yet in contrast with traditional electropolishing, it requires lower material removal for a given target roughness and makes use of mild electrolytes, for a very low negative environmental impact.

In EPPo, just like in electropolishing, material is typically thought to be removed through anodic dissolution. The high energy discharge at the interface leads however to several simultaneous phenomena limiting the rate of material removal. A gaseous vapour hull enveloping the treated surface, hallmark of plasma electrolytic processes, is an example as it limits drastically the current flowing through the cell. So is the production of soluble ions and oxides significantly modifying the effective electrolyte composition at the surface's immediate vicinity. The eventual topological properties of the treated surface are a direct result of these competing phenomena.

Careful characterisation of interfacial features thus allows to gather information on the process itself. Power spectral density analysis of height maps, obtained by laser confocal profilometry over wide areas and with varying resolutions, underline the characteristic lengths of surface features of a given surface. Comparing its results before and after EPPo, on pieces made of different materials and with varying original roughness distributions, sheds a unique light on the material removal process. Contrasting them with the results from other characterisation techniques, both *in* and *ex situ*, builds on a picture showing EPPo as a chain of processes each indispensable to the whole and to a large extent independent of one another.

**GP-11 Evaluation of the Oxidation of Cr-W-N Coating on Ferritic Steel as Bipolar Plates for Solid Oxide Fuel Cell**, *S Yang, Chi-Ju Tsan*, National University of Kaohsiung, Taiwan; *Y Chang*, National Formosa University, Taiwan; *Y Pan*, China Steel Corporation, Taiwan; *D Lin*, National University of Kaohsiung, Taiwan

Ferritic stainless steel is the one of materials of choice for SOFCs bipolar plates, because of high electrical conductivity, suitable thermal expansion compatibility, excellent mechanical properties and oxidation resistance. It has been reported that Crofer 22 H has excellent electrical conductivity and oxidation resistance with additions of Nb and W, which are better than those of Crofer 22 APU. Even though Laves phase in Crofer 22 H improves creep property, the oxidation rate is not sufficiently low to enable uncoated Crofer 22 H interconnects to meet the current 40,000 h SOFC lifetime requirement. Therefore, Crofer 22 H also requires a protective coating both to retard the oxidation rate and to prevent the volatile chromium species.

In this study, Cr-W-N coating was deposited on high-chromium ferritic stainless steel as protective coatings by using cathodic arc evaporation. Oxidation kinetics of the Cr-W-N -coated sample was evaluated through isothermal tests in an atmospheric furnace at 800 °C for 1000 h. Morphology and cross sections of scales were examined under a field-emission scanning electron microscope in both backscattered and secondary electron modes. Coating phase assemblies were assessed using X-ray diffraction. High resolution transmission electron microscopy was utilized for a close examination of the coating/alloy interfacial chemistry. After oxidation at 800 °C for 1000 h, the results showed that the dominant oxidation mechanism transfer is the growth of Cr<sub>2</sub>O<sub>3</sub> and (Mn,Cr)<sub>3</sub>O<sub>4</sub> spinel. Moreover, the doping of W benefited the formation of a  $\chi$  phase to precipitate at the oxide/coating interface, which acted as a diffusion barrier retarding the reaction rate between Cr and O.

**Bold page numbers indicate presenter**

— A —

Abrikosov, I: G6-6, 7  
 Acikgoz, C: G2-3, 5  
 Alrefae, M: G1-10, **10**  
 Arndt, M: G2-3, 5; G6-3, 7; G6-7, 8  
 Aschauer, E: G6-7, **8**  
 Auger, M: G1-8, 10  
 — B —  
 Baggio-Scheid, V: G2-1, 5  
 Banerjee, D: G3-5, 3  
 Barankova, H: G5-7, 11  
 Bardos, L: G5-7, **11**  
 Bareiss, C: G3-5, 3  
 Becker, J: G2-2, 5  
 Beganovic, N: G2-2, 5  
 Bellido-Gonzalez, V: G5-3, 11  
 Bertram, R: G6-8, 8  
 Billard, A: G2-4, 5  
 Billot, E: G2-2, 5  
 Björk, E: G6-6, 7  
 Bobzin, K: G1-1, 9; G3-4, 3; GP-8, 13  
 Bolvardi, H: G6-4, 7  
 Bork, C: G6-5, 7  
 Bort, S: GP-6, 13  
 Bradley, J: G5-5, 11  
 Brögelmann, T: G1-1, 9; G3-4, 3; GP-8, 13  
 Broughton, R: G5-2, **11**  
 — C —  
 Cabrero, J: G4-4, 1  
 Casellas, D: G3-1, 3  
 Casteletti, L: G2-1, 5  
 Chalker, P: G5-5, 11  
 Chang, W: G1-11, 10  
 Chang, Y: G4-2, 1; GP-11, 14  
 Chao, L: G4-2, **1**  
 Charlton, C: G3-5, 3  
 Chien, T: G1-11, 10  
 Chiou, A: GP-5, **13**  
 Chiu, Y: G1-11, 10  
 Collignon, P: G5-6, **11**  
 Coniglio, N: G4-4, **1**  
 — D —  
 Dambon, O: G6-2, 7  
 Dams, N: G1-4, 9  
 de Monteynard, A: G2-4, **5**  
 Delfour-Peyrethron, B: G5-5, 11  
 Dixit, S: G2-5, 5  
 Donaghy, D: G5-5, 11  
 Dosbaeva, J: G6-5, 7  
 — E —  
 Ehasarian, A: G3-11, 4  
 Eklund, P: G6-6, 7  
 El Mansori, M: G4-4, 1  
 Engels, M: GP-8, **13**  
 Eriksson, A: G6-3, **7**  
 Ertelthaler, P: G6-4, 7  
 — F —  
 Felfer, P: G6-7, 8  
 Fernandez Martinez, I: G1-4, 9; G5-3, 11  
 Fietzke, F: G1-9, 10  
 Fisher, T: G1-10, 10  
 Fontes, M: G2-1, 5  
 Fox-Rabinovich, G: G6-5, 7  
 Franz, R: GP-9, 13  
 Friedrichs, M: G6-2, **7**  
 Fukui, H: G3-7, 4

— G —

Gabriel, H: G1-4, **9**  
 Gan, K: G1-11, **10**  
 Greczynski, G: G6-6, 7  
 Greenwood, C: GP-6, 13  
 Guo, Y: G2-7, **6**  
 — H —  
 Hans, M: G6-2, 7; G6-3, 7  
 Hatton, P: GP-6, **13**  
 Hess, M: G6-8, **8**  
 Hofer-Roblyek, A: GP-9, 13  
 Hovsepian, P: G3-11, **4**  
 — I —  
 Imamura, S: G3-7, 4  
 Isomura, Y: G3-3, **3**  
 — J —  
 Jaczkowski, R: G4-1, 1  
 Jarry, O: G2-3, 5  
 — K —  
 Kacar, E: G5-8, **11**  
 Kamiyama, H: G4-5, 1  
 Karner, J: G2-2, 5  
 Kelly, P: G5-5, 11  
 Kirkpatrick, S: G5-2, 11  
 Klocke, F: G6-2, 7  
 Klostermann, H: G1-9, **10**  
 Koelker, W: G6-9, 8  
 Kohlscheen, J: G3-5, **3**  
 Kölker, W: G1-3, 9  
 Kolozsvári, S: G6-4, 7  
 Kraetzschmar, B: G1-9, 10  
 Kruppe, N: G1-1, **9**; G3-4, 3; GP-8, 13  
 Kujime, S: G3-3, 3; G3-6, **3**  
 Kulczyk-Malecka, J: G5-5, **11**  
 Kumar, A: G1-10, 10  
 — L —  
 Laugel, N: GP-10, **13**  
 Leiste, H: G1-5, 9  
 Lemmer, O: G1-3, 9; G6-9, **8**  
 Leyendecker, T: G1-3, 9  
 Li, H: G5-3, 11  
 Lin, D: GP-11, 14  
 Linke, C: GP-9, 13  
 Liu, P: G1-11, 10  
 Locks, E: G6-5, 7  
 — M —  
 Machado, D: G2-1, 5  
 Markus, M: G2-2, 5  
 Mascariñas, V: G4-3, **1**  
 Matthews, A: G2-7, 6; GP-10, 13  
 Mayrhofer, P: G6-4, 7; G6-7, 8  
 Meyer, F: G5-3, 11  
 Mezghani, S: G4-4, 1  
 Mingo, B: G2-7, 6  
 Mitterer, C: GP-9, **13**  
 Monaghan, D: G5-3, 11  
 Moser, S: G2-3, 5  
 Mráz, S: G6-3, 7  
 Mueller, A: G3-1, 3  
 — N —  
 Naderi, M: G3-4, **3**  
 Nascente, P: G2-1, **5**  
 — O —  
 Odén, M: G6-6, 7  
 Ohba, N: G3-6, 3  
 Oseguera, J: G1-7, **10**

— P —

Paiva, J: G6-5, 7  
 Pan, Y: GP-11, 14  
 Papa, F: G1-4, 9; G1-7, 10; G5-3, **11**  
 Papageorgiou, V: G1-8, 10  
 Pichler, K: GP-9, 13  
 Polcik, P: G6-7, 8  
 Poltorak, C: G1-5, 9  
 — Q —  
 Quinto, D: G4-3, 1  
 — R —  
 Ramirez, G: G3-1, **3**  
 Rees, A: GP-6, 13  
 Riedl, H: G6-4, **7**; G6-7, 8  
 Rinke, M: G1-5, 9  
 Rogström, L: G6-6, **7**  
 Ruan, D: G1-11, 10  
 Rudigier, H: G2-2, 5  
 — S —  
 Saksena, A: G6-2, 7  
 Salvador, A: G4-3, 1  
 Sanchette, F: G2-4, 5  
 Santiago, J: G1-4, 9  
 Sathrum, P: G1-2, **9**  
 Scheibe, R: G5-6, 11  
 Schiffers, C: G1-3, **9**  
 Schneider, J: G6-2, 7; G6-3, 7  
 Seid Ahmed, Y: G6-5, 7  
 Setoyama, M: G3-7, 4  
 Sgrilli, T: G5-3, 11  
 Shang, L: G2-3, **5**  
 Stolf, P: G6-5, 7  
 Strakov, H: G1-8, **10**  
 Stüber, M: G1-5, 9  
 Sze, S: G1-11, 10  
 Szyndelman, G: G2-3, 5  
 — T —  
 Takahashi, T: G3-3, 3; G3-6, 3  
 Tanaka, K: G3-7, **4**  
 Timur, S: G5-8, 11  
 Tsan, C: GP-11, **14**  
 — U —  
 Uhlmann, E: G4-1, 1  
 Ulrich, S: G1-5, **9**  
 Urgen, M: G5-8, 11  
 — V —  
 Veldhuis, S: G6-5, 7  
 Vetter, J: G2-2, 5  
 Vilaseca, M: G3-1, 3  
 — W —  
 Weißmantel, S: G6-8, 8  
 Wennberg, A: G1-4, 9  
 Winkler, J: GP-9, 13  
 Wojcik, T: G6-4, 7  
 — Y —  
 Yan, H: GP-2, **13**  
 Yang, S: GP-11, 14  
 Yelkarasi, C: G5-8, 11  
 Yerokhin, A: G2-7, 6; GP-10, 13  
 Yu, Z: GP-2, 13  
 — Z —  
 Zauner, L: G6-4, 7  
 Zemlyanov, D: G1-10, 10  
 Zhang, P: GP-2, 13