

## Surface Engineering - Applied Research and Industrial Applications

### Room Pacific E - Session G4-MoA

#### Hybrid Systems, Processes and Coatings

**Moderators:** Satish Dixit, Plasma Technology Inc., USA, Sang-Yul Lee, Korea Aerospace University, Korea (Republic of)

1:40pm **G4-MoA-1 Modelling Layered Materials Systems Using the Einstein-Hilbert Action**, Frank Papa ([frank@gpplasma.com](mailto:frank@gpplasma.com)), GP plasma, USA; T. vom Braucke, GP plasma, World Formula Apps, Canada; N. Bierwisch, Saxonian Institute of Surface Mechanics SIO, Germany; N. Schwarzer, Saxonian Institute of Surface Mechanics SIO, World Formula Apps, Germany

Complex thin film systems can be simulated by a general approach for any coating system by first characterizing it with a set of properties. Once these properties are allocated to certain aspects, we have a method to holistically simulate it. While such a holistic description is only with words, surprisingly, over 100 years ago the great mathematician David Hilbert already found a way to put a set of properties, attributes, degrees of freedom or – as all these other terms are just coding - dimensions into a mathematical formalism, it being the Hamilton extremal principle [1]. He realized that a given set of attributes is nothing else but a set of dimensions and thus, forms a mathematical space or space-time. However, Hilbert only applied his calculus to ordinary 4-dimensional space-time and to then derive the Einstein-Field-Equations [1, 2]. This methodology was further elaborated and generalized in [3, 4] providing a top-down method to describe a large complex system, that can be fed by measurement data to probe a response at various scales of a system under different scenarios. We provide a preliminary theoretical outlook toward materials science applications.

[1] D. Hilbert, Die Grundlagen der Physik, Teil 1, Göttinger Nachrichten, 395-407 (1915)

[2] A. Einstein, Grundlage der allgemeinen Relativitätstheorie, Annalen der Physik (ser. 4), 49, 769–822

[3] N. Schwarzer, "The World Formula: A Late Recognition of David Hilbert 's Stroke of Genius", Jenny Stanford Publishing, ISBN: 9789814877206

[4] N. Schwarzer, "The Math of Body, Soul and the Universe," Jenny Stanford Publishing, ISBN: 9789814968249

2:00pm **G4-MoA-2 Control of Phase Transition of VO<sub>2</sub> Films and VO<sub>2</sub>-based Terahertz and Infrared Devices**, Heungsoo Kim ([heungsoo.kim@nrl.navy.mil](mailto:heungsoo.kim@nrl.navy.mil)), Naval Research Laboratory, USA; D. Lahneman, National Research Council Fellow, USA; R. Auyeung, K. Charipar, C. Rohde, A. Pique, Naval Research Laboratory, USA

Vanadium dioxide (VO<sub>2</sub>) undergoes an insulator-metal transition (IMT) at ~67 °C, which is associated with a structural phase transition (SPT) between an insulating monoclinic phase and a metallic tetragonal phase. This SPT leads to a sharp change in conductivity and index of refraction, which make VO<sub>2</sub> an ideal material for various active devices. High quality VO<sub>2</sub> epitaxial thin films have been synthesized on various single crystal substrates with various buffer layers via pulsed laser deposition. By adjusting the growth conditions of the buffer layers, we were able to modify the interfacial strain between VO<sub>2</sub> film and buffer layer, and consequently can control the T<sub>IMT</sub> in VO<sub>2</sub> films. We have exploited the phase transition of VO<sub>2</sub> to modulate terahertz (THz) transmission by combining split ring resonator structures with phase changing VO<sub>2</sub> films. We have also demonstrated a VO<sub>2</sub>-based passive solid-state radiator for spacecraft thermal control. In this design, we were able to provide dynamic thermal emissivity control by the thermochromic phase change in VO<sub>2</sub> film, which can vary the amount of emitted power of the multilayer device. We will present details on the properties of strained VO<sub>2</sub> films and results from VO<sub>2</sub>-based devices.

This work was supported by the Office of Naval Research (ONR) through the Naval Research Laboratory basic research program.

2:20pm **G4-MoA-3 Hybrid HiPIMS and Controlled Pulsed Arc for Deposition of Hard Coatings**, Jiří Vyskočil ([jiri.vyskocil@hvm.cz](mailto:jiri.vyskocil@hvm.cz)), P. Mareš, HVM Plasma, Czechia; Z. Hubička, M. Čada, Institute of Physics CAS, Czechia

**INVITED**

A new hybrid PVD deposition system was developed based on the combination of HiPIMS pulsed glow discharge and pulsed cathodic arc discharge (HiPIMS+arc). The presence of cathodic arc discharge in the

HiPIMS is a typical situation when the glow discharge randomly transits to an arc discharge during the active part of the pulse cycle. In these typical cases, the transition appears randomly and cannot be controlled. The new hybrid HiPIMS+arc discharge works with modified pulsed high power supplies which allow the initiation of pulsed arc discharge during HiPIMS pulse at the defined time. The initiated arc during the active part of HiPIMS pulse is quenched at the end of this active part of pulse when the magnetron cathode is disconnected from the negative voltage of power supply. Several modifications with HiPIMS sequence of active pulses with arc were tested as well. The hybrid HiPIMS+arc was applied for the deposition of ta-C thin films by use of a graphite magnetron target. Carbon ta-C films were deposited on silicon substrates at different working gas pressures and different puls configurations. Deposited films were analyzed by Raman scattering and microhardness measurements and the ratio of sp<sup>3</sup>/sp<sup>2</sup> bonds was determined. The surface morphology was obtained by SEM and AFM and the presence of microdroplets was analysed for particular conditions. Achieved ta-C film parameters for this deposition source were compared with other deposition methods.

Plasma diagnostics was done in the HiPIMS+arc system by an energetically resolved ion mass spectroscopy and by a RF ion flux density monitor as both methods worked with the time resolution. Ion energy distribution functions of C<sup>+</sup>, C<sup>++</sup>, Ar<sup>++</sup>, Ar<sup>+</sup> were observed at the position of substrate for different deposition conditions in HiPIMS+arc plasma system. Measured parameters of plasma with energies of ions of carbon and argon and the values of total ion fluxes on the substrates were correlated with properties of deposited ta-C films.

# Tuesday Morning, May 24, 2022

## Surface Engineering - Applied Research and Industrial Applications

### Room Town & Country C - Session G1-TuM

#### Advances in Application Driven Research: New Methods, Materials, and Equipment for PVD, CVD, and PECVD Processes

**Moderators:** Satish Dixit, Plasma Technology Inc., USA, Martin Engels, IonBond Inc., USA

9:40am **G1-TuM-6 Photons meet Plasma – Adding Value to your Al, Mg and Ti Components**, *Anna Buling (buling@ceranod.de)*, ELB Eloxlwerk Ludwigsburg GmbH, Germany; *J. Zerrer*, ELB Eloxlwerk Ludwigsburg, Germany

PEO, plasma-electrolytical oxidation, remains as an unknown or at least niche surface technology, giving lightweight metals a hard and robust protection shell. Experiencing an increasing interest in the research society in the last decade, it is supposed to be promising for increasing lightweight potential but being not ready for wide industrial applications, due to high costs or being applicable only on small series. In this talk we will refute these reservations by unveil high demand applications of our clients, who use our surfaces on their components under harsh conditions. The CERANOD® surfaces can withstand high vibration loadings providing a high corrosion and wear protection for alloys of Al or Mg. One of our automotive clients is using our technology on highly loaded components in the power train, whereas the second generation of a larger-volume production is starting now, providing to the world's biggest OEMs. This illustrates that PEO is neither too expensive when hitting the demands nor a niche technology for very particular fields. In a further application case a Mg component modified by our ceramic atomic bonded surface found its way in large production series last year after being tested world-wide for several years in an innovative ICE application.

We will show application possibilities and different fields of our surface modification used industrial in tribological, corrosive and high temperature applications, whereas, e.g., maintenance-free power units can be realized.

Being convinced that facing the incessantly growing demands on sustainability, efficiently and endurance in automotive, aerospace and machinery applications, is only possible by lightweight, including multi-material mix, accompanied by the right solutions for the surfaces, we enhance our technologies. Most recently, we found that especially in the case of Aluminum casting alloys an adopted PEO process leads to positive tribological behavior in combination with novel low-viscosity oils. The utilization of Direct Interference Laser structuring and hybridizing with a solid lubricating polymer manifold the positive effect. A 1000-h wear test just being finished prior this submission proves a very promising solution with low friction and almost no wear.

We will report on wear and corrosion test results accompanied by SEM and EDS findings for different CERANOD® solutions. These findings will be compared and correlated with different application cases of our clients, who utilize our solutions on their end products to enable the usage of lightweight metals under harsh tribological, vibrating and /or corrosive environments, and, thus, saving a lot of energy and resources.

10:00am **G1-TuM-7 The Effect of Coating Conditions on the Life of PVD Coated Steel Rods Immersed in a Molten Aluminum Die Casting Alloy**, *Stephen Midson (smidson@mines.edu)*, *N. Delfino de Campos Neto*, *W. May*, *A. Korenyi-Both*, *M. Kaufman*, Colorado School of Mines, USA

Die casting is a high-volume casting processes, where liquid metals (primarily alloys of aluminum, zinc or magnesium) are injected at extremely high speeds and pressures into re-usable, hardened steel dies. In commercial die casting operations, PVD coatings are applied to aluminum die casting dies to reduce the erosion of the steel die by the liquid metal, and to minimize soldering (sticking) of the solidifying aluminum to the die. While these types of PVD coatings are becoming relatively widely used, there is little rigorous data to identify the best coating conditions to maximize life of the coatings. This presentation will report on a laboratory study that examined the effect of coating conditions on coating life. Three conditions have been studied, coating thickness, substrate roughness, and the impact of nitriding the steel surface prior to the PVD coating. Using an accelerated test, 11 mm diameter rods of H13 steel PVD coated with CrN were rotated at 540 rpm in a crucible of molten aluminum A380 alloy held at 700°C. The rods were periodically removed from the melt (typically every

1-2 hours) to observe their condition, and it was found that the coatings would fail either close to the melt line or at the end of the rod, and once the coated failed the underlying steel was rapidly dissolved. The results of the testing will be reported in this presentation, along with metallography characterizing the conditions and failures of the coatings.

10:20am **G1-TuM-8 Carbon-Based Surface Solutions for High Performance Forming Tools - A Journey from Material Research to Industrial Solutions**, *Vishal Khetan (vishal.khetan@oerlikon.com)*, Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein, Switzerland

**INVITED**

With rapid increase in use of non-ferrous materials especially aluminium in automotive industry, manufacturing challenges occurring from galling have become significant. Along with increase the service life of forming tools used for processing these materials, decreasing maintenance downtime and end product quality are key for good production reliability. Multiple strategies have been investigated both in academia and industry to mitigate these challenges. Some of them are reduction of surface roughness (Polishing of tools), Nitriding or Nitro carbonizing of tools (diffusion treatment) and more recently use of Carbon based coatings. Different carbon based coatings deposited by physical vapor deposition (PVD) or chemical vapor deposition (CVD) have been used for these applications. With appropriate pre and post treatment - They have helped significantly reducing galling in industry. The preferred choice of technique to deposit these coatings depends on the requirement of the application namely type and dimension of material being processed, surface roughness requirements and challenges involved in the technique to coat the tools to avoid adhesion challenges of the coatings owing to the geometry of the tools.

Owing to multiple factors involved in mitigating galling in non-ferrous materials forming - decision making to select an appropriate surface solution (a combination of coating and pre/post treatment before and after coating) needs significant trial in the form of Design of experiments or trial and error method which is time consuming and often makes decision making difficult for the OEM to select an appropriate solution in a cost effective way.

This presentation will focus on usage of a laboratory based tribological test which results in effectively evaluating adhesive wear in the system and provides appropriate insights into effectiveness of a total surface solution in an industrial application in short duration and being cost effective. The test rig presented is known as the load scanner rig which can effectively simulate the tendency of Aluminum sticking on to different substrate and suggest the effectiveness of the tooling system to provide an indication towards its effectiveness to a given application in Aluminium forming. They save time and resources needed for expensive production tests. Furthermore the tests also help the OEM or tool maker take correct decision on the type of carbon coating he needs to use for their specific alloy and tooling system. Hence, providing a pathway to maximize the cost benefit for their investment on a surface solution in a scientific way.

## Surface Engineering - Applied Research and Industrial Applications

Room Pacific E - Session G3-TuA

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

**Moderators:** Stepan Kyrsta, Oerlikon Luxembourg, Christoph Schiffers, CemeCon AG, Germany

1:40pm **G3-TuA-1 A New Tool in Coating Design: Managing Intrinsic Stresses in HiPIMS**, *Christoph Schiffers (christoph.schiffers@cemecon.de)*, T. Leyendecker, W. Kölker, S. Bolz, B. Mesic, CemeCon AG, Germany  
Until now, the residual stresses of a coating could not be adjusted independently of other properties. The classical work around was adding layers of soft materials such as CrN to the film design. Such a multilayer is not ideal when high oxidation resistance and high hardness is needed for the application.

The unique feature of HiPIMS is the ability to actively design the intrinsic stresses in a coating material. In-depth plasma analysis shows that the short HiPIMS pulses consist of several phases. Synchronizing the HiPIMS pulses on the cathodes with a pulsed Bias is the technique for attracting the ionized target species and forming the coating out of them while suppressing unwanted contributions such as gas ions.

Having control over the intrinsic stresses of a coating material by the timing of the HiPIMS synchronisation adds a fully new option to the coating designer's toolbox: stress management of the material. This triggers a paradigm shift: hardness by the composition of the material and a dense, low stress coating morphology by the HiPIMS process.

Managing intrinsic stresses in HiPIMS gives new coating options such as 12 µm thick films for heavy duty insert milling applications as well as precisely defined stresses for making TiAlSiN coatings on micro-tools even more wear resistant. Managing stresses in HiPIMS is about mastering stress and strain for the coating's application.

2:00pm **G3-TuA-2 Self-lubricating CrAlMoN High Performance Tool Coatings for Machining of TiAl6V4**, *K. Bobzin, C. Kalscheuer, M. Carlet, Nina Stachowski (stachowski@iot.rwth-aachen.de)*, Surface Engineering Institute - RWTH Aachen University, Germany; *W. Hintze, C. Möller, P. Ploog*, Institute of Production Management and Technology - Hamburg University of Technology (TUHH), Germany

Titanium alloys such as TiAl6V4 enable a significant performance improvement in different industry sectors. However, machining of titanium alloys presents a considerable challenge due to the low thermal conductivity of  $\lambda = 5.8 \text{ W/mK}$ , Young's modulus of  $110 \text{ GPa} \leq E \leq 140 \text{ GPa}$  as well as the strong adhesion tendency. This leads to high thermal and mechanical loads on the cutting edge resulting in early tool failure. Currently uncoated cemented carbide tools are commonly used for turning of TiAl6V4. However, self-lubricating physical vapor deposition (PVD) coatings like CrAlVN and CrAlMoN provide a promising approach to increase tool life. For this purpose, the coating ability to form lubricating oxide phases is essential. Self-lubricating oxide phases form under tribological loads, e.g. during machining processes due to the oxidation of specific transition metals such as vanadium or molybdenum. These oxide phases contribute to a decrease of thermal and mechanical loads in the contact between cutting edge and workpiece which may lead to an increased tool performance in TiAl6V4 cutting. In the present study, self-lubricating CrAlVN and CrAlMoN coatings were investigated on cemented carbide tools. The coatings were deposited by hybrid direct current magnetron sputtering / high power pulsed magnetron sputtering processes. Coating morphology, thickness, chemical composition, indentation hardness, indentation modulus as well as oxide phase composition were analyzed. Moreover, friction and wear behavior of the coated cutting tools were determined using a pin on disc (PoD) tribometer at  $\vartheta = 20 \text{ }^\circ\text{C}$ ,  $\vartheta = 600 \text{ }^\circ\text{C}$  and  $\vartheta = 800 \text{ }^\circ\text{C}$  against a TiAl6V4 counterpart. Additionally tool life and deformation behavior of the coated cutting inserts was analyzed after turning of TiAl6V4. The CrAlVN and CrAlMoN coatings possessed a dense morphology and a smooth surface topography. Both coating variants exhibited a good adhesion to the cemented carbide tools. For increased test temperatures, the tribological analyses showed a reduction in the coefficient of friction. In case of the CrAlVN coated samples, a friction reduction was observed at  $\vartheta = 800 \text{ }^\circ\text{C}$ . In contrast thereto, a friction reduction was already found at  $\vartheta = 600 \text{ }^\circ\text{C}$  for CrAlMoN.

As compared to the uncoated reference and to the CrAlVN coated cutting inserts an increase in tool life for CrAlMoN coated cutting inserts was achieved for turning of TiAl6V4.

2:20pm **G3-TuA-3 Coating Design for Components for Extreme Applications**, *Ricardo Alexandre (ricardo@teandm.pt)*, TEandM, Portugal  
**INVITED**

Coating design plays a paramount role in coating performance enabling performance increases in industrial components and tools. Thru the decades, coating design development has evolved from simple single layer coatings to more and more complex designs, starting from bilayers to current advanced nanostructures. When we look at extreme applications are these coatings able to protect and functionalize the surface successfully? At what extend production processes can be impacted? What about complying with demanding and restrictive product specifications? The application, at industrial scale, of sophisticated coating designs poses limitations? What kind? These are questions challenging, not only, coating developers, but also job coaters in direct contact with the market applications, where clients bring about complex and demanding surface treatment challenges driven by productivity focused production processes and sophisticated products. In order to bring some light into these questions four case studies will be presented. These case studies combine extreme working conditions (wear, corrosion, temperature, etc.) with product/process demanding specifications. Coating designs, its performance impact and industrial scale coating challenges and limitations will be discussed.

3:00pm **G3-TuA-5 The Use of Coatings to Minimize Soldering in Aluminum High Pressure Die Casting**, *Nelson Delfino de Campos Neto (ndelfino@mines.edu)*, A. L. Korenyi-Both, Colorado School of Mines, USA; C. Vian, Stellantis, USA; S. P. Midson, M. J. Kaufman, Colorado School of Mines, USA

In the aluminum high pressure die casting (HPDC) process, the molten aluminum alloy is injected into reusable steel dies at high speeds (gate speeds of between 25 and 45 m/sec) and high pressures of 70 to 100 MPa or higher. To try to prevent the liquid metal from soldering (sticking) to the steel die, lubricants (parting agents) are sprayed into the die surface prior to each shot, but some level of soldering often still occurs. In addition, the rapid cooling of the die surface by the lubricant spray can promote heat checking, thereby dramatically shortening the life of the die. During aluminum HPDC, core pins located near the gate experience extreme conditions, such as higher temperatures and greater soldering and abrasion/erosion due to interactions with the flowing liquid metal. One possible solution for minimizing soldering and to address the extreme conditions adjacent to the gate is the application of hard coatings to these more vulnerable regions of the die. However, the decision-making process for selecting appropriate coatings for application to dies is often still very subjective. Several researchers have attempted to use laboratory tests for evaluating the effectiveness of various coating materials, but these laboratory tests typically lack the high gate speeds and pressures that are inherent to the die casting process. While more complex and realistic tests have been performed using laboratory-based or production die casting machines, only a limited number of die coatings have been examined, mostly produced by the PVD process. In the study reported here, a commercial die used to produce large automotive die castings was examined, as it incorporated two core pins located directly in front of the gate. A range of PVD, PACVD and diffusion coatings were applied to the core pins, and both qualitative and quantitative measurements were used to identify the amount of aluminum soldered to each coated pin. Characterization of the soldered interfaces was performed to understand coating failure, and to indicate best die coatings for aluminum HPDC applications.

4:00pm **G3-TuA-8 Bringing Together Research, Job Coating and Market Needs**, *Carles Colominas (carles.colominas@iqs.url.edu)*, Flubetech, Spain  
**INVITED**

Job coating must be carried out through high tech but well-known technologies, to minimize technical and economic risks. A combination of high degree of specialization, customer orientation and a deep understanding of plasma technology allows PVD job coating companies to provide a proper coating service for a wide variety of applications, ranging from cutting tools to plastic or aluminum injection or biomedical implants. However, today's highly demanding and increasing market needs (local and global) push job coating companies to adapt to new circumstances to take advantage of new opportunities and finding new niches, like electric

# Tuesday Afternoon, May 24, 2022

mobility or additive manufactured parts. Research projects must be essentially applied and focused and strategically and rigorously selected to provide benefits in the short and medium-term.

In this talk, several projects developed by our SME job coating company on cutting tools, metal forming, plastic injection, energy and biomedical fields will be presented. Developments include doped amorphous carbon films and multi-layered ceramic coating systems deposited by regular magnetron sputtering, high power impulse magnetron sputtering (HiPIMS) and duplex coatings by cathodic arc deposition. In all cases, coatings were developed using industrial PVD machines. Different aspects will be discussed, including the substrate influence, adhesion to the substrate, coating microstructure control, deposition temperature, tribological properties and chemical inertness (oxidation and wettability by liquid aluminum).

4:40pm **G3-TuA-10 CrON-based Coatings for Plastic Processing Applications, Anders O. Eriksson ([anders.o.eriksson@oerlikon.com](mailto:anders.o.eriksson@oerlikon.com)), T. Vermland, D. Fopp-Spori, J. Tischhauser, Oerlikon Balzers, Oerlikon Surface Solution AG, Liechtenstein**

Polymeric materials are used in a wide range of applications including packaging, bottle caps, furniture, window frames, as well as automotive parts and high-end components. The manufacturing process often includes injection molding or extrusion processes. To achieve long lifetime, performance, and stability in high volume manufacturing processes, coatings are applied on the parts of the tooling that are in contact with the polymer melt. Increasing requirement for high-strength light-weight materials, for example driven by E-mobility, promote the use of polymers with high percentage of glass-fiber or carbon-fiber reinforcement. The use of biopolymers is also increasing. Consequently, the conditions for tools and components are becoming harsher and typically involve a combination of abrasive wear, corrosion, and plastic adhesion. To allow performance enhancements in the contemporary plastics processing industry, we describe the development of a coating solution based on CrON. The selection of coating materials, tuning of coating architecture, and coating properties will be discussed and correlated with application requirements and performance.

Preferred for Session G3

# Thursday Morning, May 26, 2022

## Surface Engineering - Applied Research and Industrial Applications

### Room Town & Country B - Session G2-ThM

#### Surface Modification of Components in Automotive, Aerospace and Manufacturing Applications

**Moderators:** Satish Dixit, Plasma Technology Inc., USA, Heidrun Klostermann, Fraunhofer FEP, Germany

8:00am **G2-ThM-1 Surface Engineering Opportunities: Harsh Environments Meeting New Strategies for Microstructural Designs (Virtual Presentation), Chris Berndt (cberndt@swin.edu.au)**, Australian Research Council, Industrial Transformation Training Centre, Australia  
**INVITED**

Harsh and extreme environments arise when the operating regime is at high temperature under corrosive conditions. Examples of such environments are numerous as industries seek greater plant efficiencies and transportation platforms strive to operate under increasingly arduous conditions. That is, engineers desire greater productivity and length of operation before refurbishment: both commercial ambitions that rest on the component lifetime.

With the above pragmatic backdrop in mind, this presentation considers adding value and performance to traditional materials by surface engineering. Examples will be drawn from the biomaterial, mining and hypersonic vehicle sectors where Surface Engineering has provided solutions that address a variety of harsh and extreme environments.

The key issue in all cases, and which connects apparently disparate applications, is 'the microstructure'. Thus, the aim of this talk is to outline the highly defective nature of the coatings that enhance the performance of components. Although the focus will be on thermal spray technology, the theme of leveraging defects for their performance attributes crosses many areas of materials science and engineering. The defective nature is controlled by the processing variables of the surfacing method. Hence, process feedback loops are necessary so that the composite-like microstructural design can be controlled.

**Acknowledgement:** This work has been supported by the Australian Research Council (ARC). The Centre for Surface Engineering for Advanced Materials is funded under the Industrial Transformation Training Centre (ITTC) scheme via Award IC180100005. Many Colleagues have contributed to this effort.

8:40am **G2-ThM-3 Plasma Nitriding of Forming Tools for the Automotive Industry - Challenges and Opportunities, Manuel Mee (manuel.mee@oerlikon.com)**, Oerlikon Balzers Coating Germany GmbH, Germany  
**INVITED**

To increase the service life of forming tools, coatings by physical vapor deposition (PVD) or chemical vapor deposition (CVD) have been used for decades. However, upscaling to ensure robust processes for large tools is difficult. In this regard, hard chrome electrodeposition has proven to be a common practice. One disadvantage in particular is the limited durability, which in the context of service life, using the example of a car body side tool, is manifested in multiple decoating and recoating involving the use of environmentally critical chemicals. In contrast, plasma nitriding is a thermochemical diffusion process for surface hardening in which the surface layer of a workpiece or component is enriched with nitrogen at low to moderate temperatures. The technology used by Oerlikon allows a loading of up to 40 tons on an area of 3x10m. Thus, an environmentally friendly surface treatment is available even for massive tools, which, compared to hard chrome, requires only a single treatment.

Nevertheless, it should be noted and taken into account that in principle a start-up of these tools have already been done beforehand and therefore process-side interactions with impurities, passivation as well as any pre- and post-treatments are possible. This applies in particular to areas hardened and welded in advance, as well as to subsequent modifications where the already nitrided surface has to be welded. Possible

consequences are material-specific and manifest themselves in tempering effects, outgassing and degradation phenomena.

The main topic of the presentation will be the requirements of users and manufacturers of forming tools and the resulting challenges. In addition to various applications and the respective restrictions with large forming tools whose mass can be far more than 10 tons, the measures derived from this to ensure process stability and quality will be discussed.

9:20am **G2-ThM-5 Enhanced Wear and Corrosion Properties of Stainless Steel by Electron Induced Plasma Nitriding, Petros Abraha (petros@meijo-u.ac.jp)**, Meijo University, Japan

In this study, we focus on recent advances in plasma source technology for materials processing applications. The plasma source used in conventional plasma nitriding treatment apply direct current, radio frequency, and microwave power between electrodes, and the potential difference accelerates electrons and ions in an electric field to generate plasma. In such a plasma source, normally, a high voltage of several kV is applied, and the generated plasma ions have high kinetic energy and large flux that bombard the surface of the sample.

Although high surface hardness can be obtained by conventional plasma nitriding treatment for austenitic stainless steel, the formation of chromium nitride reduces the chromium concentration of the base material and impairs corrosion resistance. In addition, sputtering due to ion collision also occurs, which significantly deteriorates the surface roughness. Therefore, there is a need for a nitriding method that improves the mechanical properties and corrosion resistance of austenitic stainless steel while maintaining the surface finish of the nitrided samples.

In this study, a low power plasma nitriding device with a relatively uniform plasma was used to nitride austenitic stainless steel. The results of our experiments show that the surface hardness of the nitrided samples were more than double the untreated sample while maintaining the roughness (RMS 10nm) of the mirror finished surface.

9:40am **G2-ThM-6 Tribological and Machining Performance of TiSiN(Ag) Coatings Deposited by HiPIMS, Diogo Cavaleiro (diogaocavaleiro@gmail.com)**, S. Carvalho, F. Fernandes, University of Coimbra, Portugal

Titanium alloys are one of the most common materials used in the aerospace industry. The need to machine parts in this industry poses as one of the main problems, since these alloys are well-known to be difficult to machine materials, mainly due to their low Young's modulus and poor heat conductivity. The consequent restriction in the use of high cutting speeds limits the productivity which lead to the development of self-lubricant coatings as a promising way to improve the lifetime and performance of machining tools. The main focus of this work was to investigate the effect of Ag alloying on the high temperature tribological behaviour and machining performance of TiSiN coatings. The coatings exhibit relatively high hardness (32 to 15 GPa). Tribological behaviour was assessed in pin-on-disc tests at room temperature and at 900°C, sliding against TiAl6V4 balls. Ag addition show a general decrease in the COF values across all the test temperatures. Increasing the Ag content and the temperature seems to show a beneficial effect in reducing the wear and the amount of adhered ball material. This is a promising result to reduce the built-up edge effect on the machining tests. The machining behaviour during turning of the TiAl6V4 alloy, showed that Ag additions had a beneficial effect on the machining performance of the films, especially for the higher cutting speeds. As observed for tribology, a clear reduce in the BUE was also found in machining when Ag was added to the coatings.

10:00am **G2-ThM-7 Crystal Structure, Localized Surface Plasmon Resonance and Sensing Properties of Infrared Transparent Conductive Thin Films, Liangge Xu (xuliangge@aliyun.com)**, Harbin Institute of Technology, China

Tin oxide (SnO<sub>2</sub>) has been widely explored for various applications due to its excellent n-type semiconductor properties, low resistance, and high optical transparency in the visible range. However, few studies on the preparation of SnO<sub>2</sub> films using high power pulsed magnetron sputtering have been reported. Oxygen content is a critical parameter in the practice of SnO<sub>2</sub> thin films by high-power pulsed magnetron sputtering. the average free range of Sn atoms is usually much smaller than O atoms. SnO<sub>2</sub> films deposited in a pure Ar atmosphere are likely to be oxygen-deficient and form O vacancies. and such oxygen vacancies will cause lattice distortion,

which will affect the mobility of charge carriers in the SnO<sub>2</sub> film. Therefore, oxygen is the main factor affecting the electrical conductivity of SnO<sub>2</sub> films.

In this paper, the crystal structure and infrared transparent conductive properties of SnO<sub>2</sub> films prepared at 600°C were investigated at different oxygen partial pressures. Then, it is described that integrating SnO<sub>2</sub> transparent conductive film into a multi-resonant surface enhanced infrared absorption (SEIRA) platform can overcome the shortcomings of poor selectivity and opacity of multi-gas sensing, and can simultaneously sense ultra-low concentrations of greenhouse gases on-chip. And realize the application in the transparent window scene. This strategy takes advantage of the near-field intensity enhancement (over 1500 times) of the multi-resonance SEIRA technology and the infrared light reflectivity that can be modulated by the SnO<sub>2</sub> infrared transparent conductive film. Experiments have proved that the MOF-SEIRA platform realizes synchronous on-chip sensing of VOCs, with fast response time, high accuracy, high visible light transparency, and excellent linearity in a wide concentration range. In addition, the excellent scalability to detect more gases was explored. This work opens up exciting possibilities for the realization of integrated, real-time and on-chip multi-gas detection.

10:20am **G2-ThM-8 Research on the Anti-Reflection Performance of Tetrahedral Amorphous Carbon Coatings by Ga Doping, *HoeKun Kim* ([ndkim2@naver.com](mailto:ndkim2@naver.com)), K. Lee, S. Lee, Korea Aerospace University, Korea (Republic of)**

The improvement of radiation resistance in the space solar cells(SC) is still of great importance. The main reason for space SC efficiency degradation under the action of solar wind is a reduction in carrier concentration of the base region, so that the space SC must be protected by coverglass with good protective and optical properties. However, it is very important to reduce SC weight, especially for interplanetary application. To achieve this aim relatively thin protective coating should be applied. For the decade, it has been shown that diamond-like carbon (DLC) coatings are very promising anti-reflection (AR) and protective coatings for solar cell. The advantages of DLC include high chemical stability, radiation stability and high hardness with the possibility of changing their optical properties by varying the deposition conditions. Especially, tetrahedral amorphous carbon (ta-C) coatings with extremely high hardness, smooth surface, excellent wear resistance, and better thermal stability than DLC have been paid much attention to an alternative protective coating materials. Additionally, optical properties of the ta-C coating could be improved by various metals doping. In this study, various contents of Ga were doped in the ta-C coating to improve the mechanical and optical properties of the ta-C coatings. Filtered cathodic vacuum arc (FCVA) and sputter hybrid system was co-deposited to synthesize the metal doped ta-C coating. Microstructure of the Ga doped ta-C coatings showed a columnar "moth-eye" structure that is especially useful for reducing reflections and increasing transmission between materials by the roll of light absorption. Raman spectroscopy analysis showed that all the coatings had high sp<sup>3</sup>/sp<sup>2</sup> fraction over 56%, and the hardness showed high values of 48 GPa. The ta-C coating with high Si content showed improved transmittance than other carbon based coatings, and these results indicate that the metal doped ta-C top-coating could be applied for protective & AR coating of satellite solar cell.

#### Acknowledgement

This work has supported by the National Research Foundation of Korea(NRF) grant funded by the Korea government(MSIT) (No. 2021R1A2C1010058)

## Surface Engineering - Applied Research and Industrial Applications

### Room Golden State Ballroom - Session GP-ThP

## Surface Engineering - Applied Research and Industrial Applications (Symposium G) Poster Session

**GP-ThP-1 Water and Oil Repellent Coating on Fabric Using Hollow Cathode PECVD**, *R. Mbamkeu Chakounte*, Univ Appl Sci & Arts (HAWK), Göttingen, Germany; *J. Jolibois*, AGC Interpane, Germany; *O. Kappertz*, Univ Appl Sci & Arts, (HAWK), Göttingen, Germany; **John Chambers** ([john.chambers@agc.com](mailto:john.chambers@agc.com)), AGC Plasma Technology Solutions, USA; *H. Weis*, AGC Interpane, Germany; *H. Wiame*, AGC Plasma Technology Solutions, Belgium; *W. Viöl*, Univ Appl Sci & Arts (HAWK), Göttingen, Germany

Thin film deposition is a suitable process for textile finishing at a time when environmental protection is a global concern. Thin film technology textile treatments not only avoid the harmful chemistry and resulting hazardous waste of wet chemistry, but limit the use of chemicals, water, etc., and do not require a drying system, resulting in much lower energy consumption. Various PECVD processes have therefore been developed over the years for the textile industry to overcome wet processing's disadvantages.

Water and oil repellent finishes are amongst the most studied treatments for fabrics. With PECVD, surface modification is carried out through plasma polymerization, which produces polymers with a higher degree of cross-linkage than conventional polymers. Moreover, these plasma processes are room temperature methods, so heat-sensitive monomers can be used. Among the commercially available precursors, short perfluoroalkyls can thus be used to impart water and oil repellency to fabrics, avoiding long-chain perfluoroalkyls which endanger the environment, human and animal life through the release of PFOAs and PFOS.

Within thin film industry, hollow cathode plasma source (HC) technology is increasingly gaining attention for PECVD. The key advantages of this technology are a high deposition rate and a good uniformity over large areas. However, HC is a high-density plasma source, appropriate for the deposition of inorganic layers, typically SiO<sub>2</sub>, but challenging for the deposition on fabrics without modifying their bulk properties or damaging their surface.

In this work, we demonstrate the successful use of HC technology to impart water and oil repellent properties on polyolefin textiles with fluorinated and silicone precursors. The effect of parameters such as power, pressure, gas composition and flow on water and oil repellency have been evaluated according to international standards, contact angle and the film composition analysed through FTIR measurements. Water contact angles greater than 150°, *i.e.* superhydrophobic surface, and oil repellency grade of 4 have been obtained.

Keywords: Low-pressure, hollow cathode, plasma polymerization, water and oil repellent

**GP-ThP-2 Modification of Polymer 3D Printed Parts Through Vacuum Metallization**, *Andrew Miceli* ([n00928754@unf.edu](mailto:n00928754@unf.edu)), *G. Bevill*, *S. Stagon*, University of North Florida, USA

Polymer three dimensionally (3D) printed parts have grown to become the most common prototypes for mechanical and functional design over the last decade. Like their classical injection molded counterparts, these polymer parts can benefit from surface metallization. For example, our group has metallized 3D printed polymer parts to increase mechanical performance, act as reflectors for telescopes, and protect the polymer from ultra-violet light damage. Unlike injection molded parts, the surfaces of 3D printed parts from the fused deposition modeling (FDM) method are naturally rough. Additionally, legacy wet-chemical metallization techniques for non-conductive polymers have fallen out of favor due to the use of caustic and toxic chemicals. In this presentation we demonstrate the metallization of poly-lactic-acid (PLA) 3D printed parts from the FDM method using magnetron sputtering. Prior to metallization, the parts surfaces are modified through low energy atmospheric plasma etching. Surface roughening is observed and characterized using scanning electron microscopy and laser scanning optical microscopy. Film adhesion is measured in accordance with ASTM D3359 and adhesion is shown to improve with the degree of plasma etching. Additionally, the electrical resistivity of the films is measured using four point probe. As an extension, smooth high-reflectivity surfaces are made to demonstrate the applicability

of this method for the rapid prototyping of reflectors. Overall, it is shown that sputter deposition metallization of polymer 3D printed parts is a promising technique to improve the functionality of these rapidly prototyped parts.

**GP-ThP-8 Reactive HiPIMS Deposition of AlO<sub>x</sub> Interlayer for Pt Thermistors on SiN<sub>x</sub>**, *Atasi Dan* ([atasi.dan@nist.gov](mailto:atasi.dan@nist.gov)), *E. Antunes*, *C. Yung*, *N. Tomlin*, *M. Stephens*, Applied Physics Division, National Institute of Standards and Technology (NIST), Boulder, USA; *J. Lehman*, Applied Physics Division, National Institute of Standards and Technology (NIST), USA

Thin film thermistors with negative temperature coefficient of resistance (TCR), like Pt, are desirable for temperature-sensing applications. To achieve high sensitivity in detecting a small change in temperature, a high-quality interlayer of AlO<sub>x</sub> is required between the SiN<sub>x</sub> membrane and the Pt thermistor. High power impulse magnetron sputtering (HiPIMS) is known to produce high-quality thin films by generating high ionization of sputtered material which can significantly improve properties of the film over conventional sputtering techniques. In the case of reactive HiPIMS, it is important to monitor the reactive gas flow, peak current, growth rate, etc, to avoid instability in the process and control the growth of the poisoned layer on the target surface.

In this study, we investigate how target poisoning on the Al surface in the presence of oxygen can be influenced by a change in pulse length or frequency. We also show that an appropriate selection of deposition parameters can systematically provide an easier control in the reactive HiPIMS process to determine the performance of the film. The present results open the possibility of using a HiPIMS-based AlO<sub>x</sub> interlayer in Pt/AlO<sub>x</sub>/SiN<sub>x</sub> thermistors for achieving a high negative TCR. Additionally, we show the role of Pt target power in enhancing the TCR of Pt/AlO<sub>x</sub>/SiN<sub>x</sub>.

**GP-ThP-9 Synthesis of Large Area ta-C Coating by Single-bend FCVA Source Using in-line PVD System**, *HoeKun Kim* ([ndkim2@naver.com](mailto:ndkim2@naver.com)), *K. Lee*, *S. Lee*, Korea Aerospace University, Korea (Republic of); *J. Kim*, University of Incheon, Korea (Republic of)

Tetrahedral amorphous carbon (ta-C) coating is a hydrogen-free carbon coating with the remarkable properties comparable with those of diamond film, such as high hardness, optical transparency and chemical inertness. Moreover, ta-C coating can be synthesized through a relatively convenient method and has a much smoother surface, making the tribological performances of ta-C coating better than those of other diamond coatings. Among the various attempts used to prepare ta-C coatings, the filtered cathodic vacuum arc (FCVA) method is a particularly suitable technique for the mass-production of industrial ta-C coatings, and the performable properties make ta-C coatings suitable for potential commercially important components in applications such as automobile accessories, optical devices, and aerospace parts. In this study, large area ta-C coating on a 300x300mm STS plate was synthesized by single-bend filtered cathodic vacuum arc (FCVA) using in-line PVD system. Source and bend filter connecting 45° bent together were used to produce carbon plasma from a graphite target with a diameter of 50mm and a purity of 99.99%. Especially, raster magnet system was designed and constructed for large area synthesis in this source. The large area ta-C coatings with 1.8μm thickness were synthesized successfully, and thickness uniformity was showed as 92.4%. Raman spectroscopy analysis showed that the ta-C coatings had high sp<sup>3</sup>/sp<sup>2</sup> fraction over 63%, and the hardness showed high values of 48.5 GPa. In addition, the ta-C coatings with 700nm in thickness, a sp<sup>3</sup>/sp<sup>2</sup> fraction over 74%, and about 63 GPa hardness could be synthesized with a similar uniformity. Detailed experimental results will be presented.

### Acknowledgement

This work has supported by the National Research Foundation of Korea(NRF) grant funded by the Korea government(MSIT) (No. 2021R1A2C1010058)

## Author Index

### Bold page numbers indicate presenter

— A —

Abraha, P.: G2-ThM-5, **5**  
Alexandre, R.: G3-TuA-3, **3**  
Antunes, E.: GP-ThP-8, **7**  
Auyeung, R.: G4-MoA-2, **1**

— B —

Berndt, C.: G2-ThM-1, **5**  
Bevill, G.: GP-ThP-2, **7**  
Bierwisch, N.: G4-MoA-1, **1**  
Bobzin, K.: G3-TuA-2, **3**  
Bolz, S.: G3-TuA-1, **3**  
Buling, A.: G1-TuM-6, **2**

— C —

Čada, M.: G4-MoA-3, **1**  
Carlet, M.: G3-TuA-2, **3**  
Carvalho, S.: G2-ThM-6, **5**  
Cavaleiro, D.: G2-ThM-6, **5**  
Chambers, J.: GP-ThP-1, **7**  
Charipar, K.: G4-MoA-2, **1**  
Colominas, C.: G3-TuA-8, **3**

— D —

Dan, A.: GP-ThP-8, **7**  
Delfino de Campos Neto, N.: G1-TuM-7, **2**;  
G3-TuA-5, **3**

— E —

Eriksson, A.: G3-TuA-10, **4**

— F —

Fernandes, F.: G2-ThM-6, **5**  
Fopp-Spori, D.: G3-TuA-10, **4**

— H —

Hintze, W.: G3-TuA-2, **3**

Hubička, Z.: G4-MoA-3, **1**

— J —

J. Kaufman, M.: G3-TuA-5, **3**  
Jolibois, J.: GP-ThP-1, **7**

— K —

Kalscheuer, C.: G3-TuA-2, **3**  
Kappertz, O.: GP-ThP-1, **7**  
Kaufman, M.: G1-TuM-7, **2**  
Khetan, V.: G1-TuM-8, **2**  
Kim, H.: G2-ThM-8, **6**; G4-MoA-2, **1**; GP-ThP-  
9, **7**  
Kim, J.: GP-ThP-9, **7**  
Kölker, W.: G3-TuA-1, **3**  
Korenyi-Both, A.: G1-TuM-7, **2**

— L —

L. Korenyi-Both, A.: G3-TuA-5, **3**  
Lahneman, D.: G4-MoA-2, **1**  
Lee, K.: G2-ThM-8, **6**; GP-ThP-9, **7**  
Lee, S.: G2-ThM-8, **6**; GP-ThP-9, **7**  
Lehman, J.: GP-ThP-8, **7**  
Leyendecker, T.: G3-TuA-1, **3**

— M —

Mareš, P.: G4-MoA-3, **1**  
May, W.: G1-TuM-7, **2**  
Mbamkeu Chakounte, R.: GP-ThP-1, **7**  
Mee, M.: G2-ThM-3, **5**  
Mesic, B.: G3-TuA-1, **3**  
Miceli, A.: GP-ThP-2, **7**  
Midson, S.: G1-TuM-7, **2**  
Möller, C.: G3-TuA-2, **3**

— P —

P. Midson, S.: G3-TuA-5, **3**  
Papa, F.: G4-MoA-1, **1**  
Pique, A.: G4-MoA-2, **1**  
Ploog, P.: G3-TuA-2, **3**

— R —

Rohde, C.: G4-MoA-2, **1**

— S —

Schiffers, C.: G3-TuA-1, **3**  
Schwarzer, N.: G4-MoA-1, **1**  
Stachowski, N.: G3-TuA-2, **3**  
Stagon, S.: GP-ThP-2, **7**  
Stephens, M.: GP-ThP-8, **7**

— T —

Tischhauser, J.: G3-TuA-10, **4**  
Tomlin, N.: GP-ThP-8, **7**

— V —

Vermland, T.: G3-TuA-10, **4**  
Vian, C.: G3-TuA-5, **3**  
Viöl, W.: GP-ThP-1, **7**  
vom Braucke, T.: G4-MoA-1, **1**  
Vyskočil, J.: G4-MoA-3, **1**

— W —

Weis, H.: GP-ThP-1, **7**  
Wiame, H.: GP-ThP-1, **7**

— X —

Xu, L.: G2-ThM-7, **5**

— Y —

Yung, C.: GP-ThP-8, **7**

— Z —

Zerrer, J.: G1-TuM-6, **2**