Surface Engineering - Applied Research and Industrial Applications

Room Sunrise - Session G2

Components Coatings

Moderators: Kenji Yamamoto, Kobe Steel Ltd., OsmanL. Eryilmaz, Argonne National Laboratory, USA, Jolanta Ewa Klemberg-Sapieha, Polytechnique Montreal

1:50pm **G2-2 Advanced Metal/Ceramic Nano-multilayers for Joining Applications: Interplay between Nano-confinement, Stress Relaxation and Environmental Conditions**, ***Mirco Chiodi***, *C Cancellieri, F Moszner,* Empa, Laboratory for Joining Technologies & Corrosion, Switzerland*; M Andrzejczuk,* Warsaw University of Technology, Poland*; J Janczak-Rusch, L Jeurgens,* Empa, Laboratory for Joining Technologies & Corrosion, Switzerland

The industrial demand to manufacture complex, heterogeneous devices has grown exponentially. Such devices typically comprise various materials with different heat sensitivities and thermal expansion coefficients. Thus, novel approaches towards joining of complex multi-materials at ever-reduced temperatures are emerging. Among the others, a promising strategy involves the use of nanostructured brazing fillers in the form of coatings consisting of Nano-MultiLayers (NMLs) of metallic brazing filler and a chemically inert barrier. The interplay between spatial confinement, internal stress gradients and the processing environment can stimulate phase-transitions and/or enhanced kinetics associated with a significant outflow of the confined metallic brazing filler to the surface at reduced temperatures. This phenomenon could be exploited for joining materials well below the melting point of the bulk constituents.

Here, we present a comprehensive investigation of the microstructural evolution of (Ag/AlN)5nm/10nm NML coatings upon heating in air. SEM/TEM results evidence the strong migration of Ag from the inner part of the NML to the surface. Silver particles as large as 1 µm are found after a heat treatment in air up to 420 °C. XRD characterization and pole figures confirm that Ag and AlN are initially strongly textured. The in-plane texture is partially lost upon heating in air, as a consequence of the Ag migration and the partial oxidation of AlN. The microstructural evolution of the Ag/AlN NML during annealing was monitored by real-time XRD collected at the synchrotron. Beyond this temperature, a strong increase in the Ag coherency domain is registered. Such increase correlates with the Ag particle appearing on the surface and subsequently coarsening. The average stress state in the Ag layers has been qualitatively evaluated using the real-time XRD data. The results indicate an accumulation of (thermal) stress between 200-280 °C which is then released at higher temperatures, triggering the massive Ag migration. Identical experiments carried out in vacuum or in absence of multilayered structure indicate that no Ag migration takes place. To elucidate the crucial role of oxygen on the Ag mobility at low temperatures, an extensive XPS analysis was carried out on samples heated at different temperatures (from 200 °C to 420°C). The results indicate that oxygen is penetrating through the NML structure and (partially) reacting with both AlN (forming AlOx) and Ag (being adsorbed and/or incorporated at Ag layers surface). The adsorption and dilution of oxygen in Ag can strongly enhance its atomic mobility, thus further easing its relocation on the NML surface.

2:10pm **G2-3 Coatings for the Aerospace Industry**, ***Jeffrey Lince***, The Aerospace Corporation, USA **INVITED**

Coatings are of critical importance to both aircraft and spacecraft. However, environmental requirements for coatings between the two vary considerably. Coatings used to protect turbojet engines must operate in air at a wide range of temperatures from ambient to greater than 1200°C. In contrast, coatings used on spacecraft may be required to achieve their function in air during prelaunch storage, and also on orbit, i.e., in vacuum, at lower temperatures, and in a potentially radiation-rich environment. This talk will provide a survey of coatings techniques for aircraft and spacecraft, concentrating on areas where improvements are needed. For example, superalloys are used to maintain strength at elevated temperatures in aircraft applications, but compatible coatings are required to form thermal barriers, and to minimize corrosion and fatigue. Thermal spray coatings are being used extensively for this purpose. In addition, thermal spray and PVD coatings are used to coat ceramic fiber cloth with metals to form advanced metal-matrix composites (MMCs) that are low weight and exhibit superior materials properties. Solid lubricating/antiwear coatings in aircraft and space applications often involve different materials: CaF_2 and metal oxides provide low friction and wear in air at elevated temperatures, while MoS_2 is preferred at low temperatures in vacuum. Chromate coatings are used for corrosion protection on both aircraft fuselages and spacecraft surfaces, but improved performance can be met with more modern coating materials like rare earth salts, sol gel organic-inorganic composites, and resin composites. In this talk, current research being done in order to push coating performance to meet continually increasing requirements will be discussed.

2:50pm **G2-5 Triboactive CrAlN+X Hybrid dcMS/HPPMS PVD Nitride Hard Coatings for Friction and Wear Reduction on Components**, *K Bobzin, T Brögelmann,* ***Christian Kalscheuer***, Surface Engineering Institute - RWTH Aachen University, Germany

Increasing environmental awareness and energy costs are major driving forces behind the development of energy efficient machines. Simultaneously, increased energy efficiency often leads to higher power densities. The consequences are load spectra which often exceed the load carrying capabilities of the base material and therefore higher wear rates and reduced life times. Therefore, coatings for the application on highly loaded components were developed. Besides diamond-like carbon (DLC) coatings, nitride hard coatings deposited by physical vapor deposition (PVD) show a high potential for wear reduction on machine components. However, regarding friction reduction in lubricated tribological contacts, nitride hard coatings still exhibit a high demand for research since state-of-the art lubricants are tailored to interact with steel surfaces in order to form friction reducing tribolayers. Therefore, the addition of tribo effective elements (X) into nitride hard coatings is a promising approach to enhance tribological interactions with lubricants and to reduce friction. In order to deposit PVD coatings on complex geometries with increased mechanical properties, the high power pulsed magnetron sputtering (HPPMS) technology shows high potential. The aim of the paper is the analysis of the tribological interaction between the nitride hard coating (Cr,Al)N+Mo and lubricants. Therefore, a mineral base reference oil and a mineral oil doped with a sulphur additive were investigated regarding interactions with the coatings under tribological conditions. The coatings were deposited in a low temperature T ≤ 200 °C hybrid PVD coating process on case hardened steel AISI5115 (16MnCr5E). Hybrid PVD coating processes allow the combination of direct current magnetron sputtering (dcMS) and HPPMS. The coating and compound properties were investigated. The tribological behavior of the coatings was tested in a pin on dics (PoD) tribometer against inert ceramic Si_3N_4 counter bodies to ensure that tribologial interactions can only occur due to reactions between the coatings and the lubricant. The uncoated case hardened steel AISI5115 (16MnCr5E) and a (Cr,Al)N coating were investigated as reference. The tests were conducted at temperatures T = 90 °C and T = 130 °C at Hertzian contact pressures $p_H \approx$ 1,600 MPa and $p_H \approx$ 1,900 MPa. Under the given tribological conditions a friction reduction was achieved by adding the tribo effective element Mo into the coatings. Raman spectroscopy revealed that MoS_2 was formed in-situ during the tribological tests. The investigated (Cr,Al)N+Mo coatings are therefore a promising approach for friction reduction in highly loaded tribological systems.

3:10pm **G2-6 Tribological Performance of PTFE Based Composite Seal Materials Against Diamond Like Carbon and Catalytically Active Nitride Based Nano-composite Coatings**, ***OsmanL. Eryilmaz***, *G Ramirez, A Erdemir,* Argonne National Laboratory, USA

Natural gas (NG) consumption has been grown rapidly during the last decade, and NG production is projected to further increase by 44% through 2040. Consequently, the potential for methane emission (which can deter GHG benefits of using NG) is also expected to increase throughout the supply chain. Accordingly, the NG industry is facing tough challenges toward mitigating methane emissions in the form of not only adopting new low-emission gas compression technologies but also upgrading or enhancing the currently installed compressors in the field. NG industry uses two types of compressors in the production, delivery and storage of NG. Most common one is the reciprocating NG compressors which account for the largest amount of leakage of methane. Primary leak source of those compressors are wear and scratches on rod and seal material surfaces in piston rod packing systems. One approach is to apply hard coatings onto rod surfaces to prevent wear on the rod side, however it could be detrimental on the delicate Teflon based counter face seal side.

New coatings and surface modification techniques are in need to prevent wear on both surfaces. One approach would be to develop a hard coating that generates tribo-films that beneficial to Teflon based seal side as well,

this would minimize wear on both sliding surfaces, or another approach would be to use already known solid lubricant coating such as diamond like carbon. Accordingly, in this paper, we concentrate on the friction and wear performance of DLC, and catalytically active nano-composite nitride based coatings against PTFE type seal materials filled with different fillers (Carbon, MoS2, glass, etc.). A series of vanadium nitride – copper nano-composite, and hydrogenated DLC films were prepared using high power impulse magnetron sputtering (HPIMS). The films were grown on 52100 steel substrates for tribological tests. X-Ray Diffraction (XRD), raman Spectroscopy, nano-indentation techniques were used to characterize the structural, mechanical and chemical nature of the resultant coatings. Bench-top tribological tests were conducted by using oil lubricated reciprocating test rig. Overall, the effect of sliding speed, contact pressure, type of PTFE fillers on the tribolocigal behavior of coatings were investigated. Both nitride and carbon based coatings improved the wear performance of the system depending on the type PTFE composite counter-face used. At the end of each test, confocal raman was used to evaluate sliding interfaces and any structural changes resulting from the tribological tests to shed more insight into the possible mechanisms responsible.

Surface Engineering - Applied Research and Industrial Applications
Room Sunrise - Session G6

Application-driven Cooperation between industry and Research Institutions

Moderators: Hamid Bolvardi, Oerlikon Balzers, Oerlikon Surface Solutions AG, Kirsten Bobzin, Surface Engineering Institute - RWTH Aachen University, Germany

3:30pm **G6-7 Research Behind a High Performance Metal Cutting Tool***,* ***Jacob Sjölén****,* SECO Tools, Sweden **INVITED**

The research and development of metal cutting tools has over the last century led to incredible improvements in manufacturing. Cost efficient solutions, more advanced materials and super-alloys are key-words in a modern machine shop, meaning demands on increased productivity and new solutions for machining of difficult materials. This, in turn, generates extreme conditions at the cutting tool edge, comprising e.g. high pressure, high thermal load and chemical interaction.

To meet the demand of endurance at these circumstances, there is a need for a deep material understanding combined with the continuous development of material properties, tool design and cutting process. Thus, this requires combined efforts over the entire R&D-chain covering activities from fundamental research to field testing, including co-operations over the borders between academia and industry and compiling all this knowledge into a High Performance Metal Cutting Tool.

This work will present examples of how such collaboration between academia and industry leads to an increased understanding of existing conditions, improved productivity of new tools and technology platforms for the development of next generation metal cutting tools. More specifically, how application driven research of the wear properties in the metal cutting processes connects to the thermal, chemical and mechanical behavior, which further relates to synthesis, composition and structure of materials used for metal cutting tools, for detailed understanding of the whole system in order to continuously push the limits for the tools.

4:10pm **G6-9 Residual Stress Measurement Technique for Static and Dynamic Coating Processes using Micro-machined Stress Sensors for Scientific and Industrial Applications***, L Banko,* ***Dario Grochla****, A Ludwig,* Ruhr-Universität Bochum, Germany

The mechanical behavior of the thin film-substrate compound strongly correlates with residual stress especially with respect to the hardness, adhesion and tribological performance. The film stress is affected by different contributions like interfacial stress or different coefficients of thermal expansion as well as dislocations, impurities, voids and grain boundaries. Several stress components are related to the film´s microstructure and can indicate the presence of phases. Failure mechanisms like buckling or cracking often can be ascribed to intrinsic and/or extrinsic film stress.

Quantitative determination of thin film stress can be challenging, especially for very thin films, e.g. optical or barrier coatings < 100 nm. XRD strain measurement might fail because of low intensity or texture. Curvature stress measurements base on the Stoney equation to calculate the stress knowing the substrate thickness, film thickness and the radius of curvature. However, in thin coatings the bending force might not be strong enough to deflect standard available substrates that are usually 200 – 500 µm thick. The appropriate selection of the substrate thickness is the key to control the sensitivity of the curvature method. By adequate tuning of the substrate thickness a high measurement resolution (few MPa) can be obtained in any film thickness range.

Micro-machined cantilever stress sensors (6.55 x 7.4 mm^2) have been developed to meet this criterion. Sensors were fabricated with a substrate thickness in the range of 30 – 120 µm. The Sensors were validated in several research and industrial type plasma-based depositions with film thicknesses ranging from < 10 nm to 3 µm and have been applied in static as well as dynamic coatings processes. The small sensors allow localized stress determination in different places of the deposition chamber. The combination of the stress sensor and an optical holography test stand, provides a fast and easy point-and-click measurement to quantify residual stress. Additionally, the coefficient of thermal expansion can be calculated by thermal cycling of the sensors on a heating platform as the Young's modulus is known. Furthermore, it will be shown, how residual stress measurements can help to understand different thin film behaviors and reveal non-uniformities in the coating processes. The stress sensors can be used as an indicator (figure of merit) in industrial deposition processes, to easily monitor thin film quality over long time periods.

Acknowledgment: SFB-TR 87

4:30pm **G6-10 Improvement of Thermal Stability and Oxidation Resistance of Molybdenum Nitride***,* ***Fedor F. Klimashin****,* CDL-AOS TU Wien, Austria*; M Arndt,* Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein*; P Polcik,* Plansee Composite Materials GmbH, Germany*; H Euchner, N Koutná,* TU Wien, Austria*; D Holec,* Montanuniversität Leoben, Austria*; P Mayrhofer,* TU Wien, Austria

The ever-growing industry demands challenge researchers to develop ever-better performing materials. Evidently, the industry–university collaboration, implying funding for the research activities and monetization of the developed cutting-edge technologies, is mutually beneficial, and one of the most effective ways of engaging the knowledge exchange between industry and research institutions is a direct research collaboration of both.

The present research – conducted in cooperation with Oerlikon Balzers AG and Plansee Composite Materials GmbH – aimed at improving thermal stability and oxidation resistance of the cubic-structured molybdenum nitride γ-MoN_x. The refractory metal Mo is successfully used as alloying element (for nitrides in particular) for the purpose of improving hardness[1], toughness[2,3], friction coefficient[4], while the application of γ-MoN_x – though high-temperature allotropy – is strongly limited to 300–500 °C unless an oxygen-free atmosphere is provided. Optimizing the composition of alloying elements (able to form a dense oxide scale at elevated temperatures, e.g. Al and Cr) and vacancies (inherent in Mo-based nitrides), superhardness (over 40 GPa) combined with high resistance to plastic deformation (~0.4 GPa) and significantly improved thermal stability and oxidation resistance (both > 900 °C) could be achieved.

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3 Zhou, L., David, H. & Mayrhofer, P. H. Ab initio study of the alloying effect of transition metals on structure, stability and ductility of CrN. *J. Phys. D: Appl. Phys.* **46**, 365301 (2013).

4 Glatz, S. *et al.* Influence of Mo on the structure and the tribomechanical properties of arc evaporated Ti-Al-N. *Surface and Coatings Technology* (2017).

4:50pm **G6-11 Empirical Alloys-by-design Theory Calculations to the Microstructure Evolution Mechanical Properties of Mo-doped Laser Cladding NiAl Composite Coatings on Medium Carbon Steel Substrates***, C Lin,* ***Wei-Yu Kai****,* National Taipei University of Technology, Taiwan

An experimental investigation is performed into the microstructure and mechanical properties of Mo-doped (0, 3, 6, 9,12 and 15wt% Mo) NiAl coatings deposited on medium carbon steel substrates with a laser cladding process. It is shown that as the Mo content increases, the density

of the Mo-rich phase network structure increases and the grain size of the laser-clad coating reduces. Furthermore, the hardness and wear test results indicate that a higher Mo addition not only increases the strength of the NiAl coating, but also improves its toughness and wear resistance. On the other hand, the underlying mechanism of phase formation and elements diffusion has been firstly studied by first principle simulation and DICTRA corresponding to the SEM micrographs of cross-section and coating-substrate interface. It is presented that pure NiAl and Mo phase exist a lowest formation enthalpy complying with the results of Thermocalc simulation. What more, the diffusion of Fe from substrate controlled by the addition of refractory element of Mo and the solid or liquid state of coatings during cladding process. Thus, with a combination of alloy design, the role of laser cladding process, and the theoretical calculation, this study provides a potential guideline for evolution and practical application of intermetallic compound in the future. In general, with a combination of alloy design, the role of laser cladding process, and the theoretical calculation in this study provide a useful source of reference for extending the applications of NiAl composite coatings in the aerospace field and other advanced industries.

Surface Engineering - Applied Research and Industrial Applications

Room Sunrise - 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

8:40am **G4-3 Nitriding and DLC Coating of Aluminum Alloy Using High Current Pressure-Gradient-Type Plasma Source**, ***Akio Nishimoto***, Kansai University, Japan; *E Furuya, K Kousaka,* Chugai Ro Co., Ltd., Japan

The low hardness and poor tribological performance of aluminum alloys restrict their wide applications in automotive fields. However, protective hard coatings deposited onto aluminum alloys are effective for overcoming their poor wear properties. In this study, a diamond-like carbon (DLC) film followed by a nitriding layer was deposited onto an aluminum alloy via a pressure-gradient-type plasma source using nitrogen and acetylene gases. This pressure-gradient-type plasma source is operated at a low discharge voltage of 60-100 V and a high current of 60-130 A. An aluminum alloy EN AW-5052 sample was plasma nitrided for 4 h at 520 ºC under 0.09-1.1 Pa. DLC was then coated with an acetylene gas after plasma nitriding using the same apparatus. The Vickers microhardness of the surface nitrided at 0.51 Pa reached approximately 340 HV from 125 HV. In addition, glow discharge optical emission spectrometry (GD-OES) revealed that nitrogen was concentrated at the surface region. After the DLC coating, the sample was reddish brown in color. GD-OES results demonstrated that a carbon-rich region formed in the top surface region (DLC film), followed by the formation of a nitrogen-rich region (nitriding layer). Nanoindentation test showed that the hardness of the top surface (DLC film) was 10.3 GPa. The DLC coating also exhibited good tribological performance in a ball-on-disk wear test, with friction coefficients of approximately 0.17, which was characterized as low value of DLC. In addition, aluminum nitride (AlN) interlayer was deposited on the nitriding layer by ion plating method in order to enhance adhesion between the DLC film and the substrate. Rockwell indentation indicated good adhesion. Hardness, roughness, and structure of the DLC film deposited on the AlN interlayer was investigated.

9:00am **G4-4 Towards Hard yet Tough Ceramic Coatings**, ***Sam Zhang***, Nanyang Technical University, Singapore

Over the past decades, hard and super hard ceramic coatings have been developed and widely used in various industrial applications. Meanwhile, an increasing number of studies have realized that the toughness is just as crucial, if not more, than hardness especially for ceramic coatings. However, hardness and toughness do not go naturally hand in hand. In other words, hard coatings usually are brittle and less durable while toughened coatings are of lower strength. For practical engineering applications, it is more desirable to have coatings with high hardness without sacrificing toughness too much. In this talk, a review is presented on continuous progress to realize hard-yet-tough ceramic coatings from an angle of hardening as well as toughening.

9:20am **G4-5 Flash Lamp Annealing (FLA) for Post-deposition Treatment at High Throughput**, ***Thoralf Gebel***, University of Applied Sciences Mittweida, Germany; *M Neubert,* ROVAK GmbH, Germany; *W Skorupa,* Helmholtz Zentrum Dresden-Rossendorf, Germany **INVITED**

Today's deposition processes for large area / large volume applications are strongly influenced by cost saving issues, by environmental regulations and by aspects of improved throughput. Especially in the case of thermal processing steps (e.g. for transparent conductive oxide (TCO) layers for displays on flexible glass) this becomes a very important aspect: substrates which need to be heated during or after the deposition process require sophisticated handling systems, and the moving substrates heated to elevated temperatures require cooling zones which may limit the throughput. A promising approach to overcome these problems are novel ultra-short time thermal processes with treatment times in the (sub)millisecond range. By using pulsed photonic treatment (e.g. by means of laser or flash lamp annealing) only the surface layers of the substrate materials are heated and therewith the thermal budget is strongly reduced. The work presented here focuses on investigations of such new thermal processes by using simulations and providing energy & cost saving models. Aspects of process design and overlapping issues and their influence to homogeneity will be discussed.

10:00am **G4-7 Evaluating the Effect of Titanium-Based PVD Metallic Thin Films on Nitrogen Diffusion Efficiency in Duplex Plasma Diffusion/Coating Systems**, ***Gorkem Yumusak***, *A Leyland,* University of Sheffield, UK; *A Matthews,* University of Manchester, UK

Titanium is a very popular engineering metal due to its outstanding properties, such as low density and high specific strength. However, the wear resistance of titanium is very poor in many industrial environments. Wear resistant hard coatings can be used to increase the service lifetime of manufactured products but the effectiveness of these coatings on titanium is sometimes weak due to poor load bearing capacity of the substrate. Therefore, titanium alloys need certain pre-treatments before the Physical Vapour Deposition (PVD) of ceramic hard coatings.

In this work, triode plasma nitriding (TPN) has been applied in order to increase the load bearing capacity of titanium alloys. It is known that the adhesion between titanium alloy substrates and PVD hard coatings can be increased significantly after substrate diffusion treatment [1]. TPN treatments were used in this work because the diffusion of the nitrogen can be achieved more easily at lower temperatures and shorter times, without the need for hydrogen in the gas mixture.

The efficiency and effectiveness of triode-plasma diffusion treatment can be increased by applying a thin PVD metallic layer on titanium alloy substrates, before plasma nitriding [2, 3]. In this context, different compositions of β-titanium coating (stabilized by addition of Nb) were produced on α+β Ti-6Al-4V and β Ti-15Mo substrate materials; the formation of β phase in Ti-Nb coatings before nitriding (and of nitride phases after TPN treatment at 500-700ºC) was analysed.

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10:20am **G4-8 Properties of Surface Passivation at Si/Al_2O_3 Interface Annealed in Different Gas Ambient**, *C Yang,* National Chung-Hsing University, Taiwan; ***Chun-Wei Huang***, *C Hsu,* Da-Yeh University, Taiwan; *C Kung,* National Chung-Hsing University, Taiwan; *S Lien,* Da-Yeh University, Taiwan; *W Zhu, X Meng, X Zhang,* Xiamen University of Technology, China

Efficient surface passivation is crucial in most electronic devices, especially in solar cells, where the generated electron-hole pairs need to be collected by contacts before recombining at the surfaces. Aluminum oxide (Al_2O_3) film synthesized by atomic layer deposition system (ALD) offers a high level of surface passivation for p-type passivation emitter and rear cells (PERC). The high passivation quality of Al_2O_3 is related to high negative charge combined with low interface density. The post-annealing process after the deposition of Al_2O_3 films can effectively activate the passivation. The objective of this work is to optimize the passivation performance by hiring different annealing temperature and atmosphere containing air, nitrogen, oxygen, and forming gas (95 % N_2+5 % H_2) inside the furnace.

The silicon wafers used in this study is mono-crystalline p-type commercial-grade CZ silicon wafers with a thickness of 200±20 µm and a resistivity of 0.5 to 5 Ω-cm. The original lifetime of bare wafer is under 5 µs. Initially silicon wafers are cleaned through a standard Radio Corporation of America (RCA) cleaning process and textured using 6 % KOH solution. After that, the uniform 25 nm Al_2O_3 films are prepared by non-vacuum spatial atomic layer deposition on double sides of wafers. Post annealing process in air, nitrogen, oxygen, and forming gas ambient are performed to the samples at 350°C to 650°C for 30 min using a furnace. To characterize the passivation quality of Al_2O_3 films, effective carrier lifetime (τ_{eff}), negative fixed charge (Q_f) and interface trap density (D_{it}) are measured and determined. The result shows that as the annealing temperature increasers, the τ_{eff} increases first and then decreases after peak τ_{eff} of around 152 µs at 450°C. It indicates that appropriate annealing temperature can activate the passivation effect of Al_2O_3, but τ_{eff} may decline rapidly when the temperature over 600°C due to the crystallization of Al_2O_3. The trend of τ_{eff} value can be further explained by the fixed negative charge and D_{it}. The highest Q_f of -1.07×10^{12} cm^{-2} and lowest D_{it} of 7.5×10^{12} $eV^{-1}cm^{-2}$ that can effectively reduce recombination on the Si/Al_2O_3 interface are determined from the capacitance-voltage curve at 450°C, hence increasing the lifetime of carriers before trapped by defects. For gaining higher τ_{eff}, the samples are annealed in various atmosphere for 450°C. The better τ_{eff} of around 180 µs is acquired while annealed in forming gas. This is probably attributed to the extra chemical passivation

effect via filling dangling bonds on the Si surface by hydrogen atom. The results of this study can able be used for high efficiency PERC cells.

10:40am **G4-9 High Performance Solar Selective Coatings based on TiNxOy**, ***Cho-Yen Lee***, *J Ting,* National Cheng Kung University, Taiwan

In this work, a series of TiNxOy films have been investigated for used as solar selective absorbers due to their remarkable optical, mechanical, and electronic properties. The films were deposited using a reactive magnetron sputtering technique. A pure titanium target was used and the deposition took place in different mixtures of oxygen, nitrogen, and argon. The obtained TiNxOy was then coated with an anti-reflection layer, consisting of metal or non-metal oxides with desirable refractive index. The TiNxOy films and the resulting multilayer coatings were analyzed for the material characteristics. Effects of the material characteristics on the optical performance is presented and discussed. We demonstrate that the resulting multilayer coatings make a breakthrough on the limitation of traditional absorbers' monotonous color appearance which is expected to be desirable in many applications.

11:00am **G4-10 Diagnostics of Surface Roughness during Electrolytic Plasma Polishing Pre-treatment for Stainless Steels**, *V Mukaeva, E Parfenov, R Farrakhov, M Gromova,* Ufa State Aviation Technical University, Russian Federation; ***Aleksey Yerokhin***, The University of Manchester, UK

Pre-treatments of steel surface constitute an important step for successful physical vapor deposition of commercially used protective coatings such as TiN, Ti and other. Electrolytic plasma polishing (EPPo) provides several effective pre-treatment operations such as deburring, cleaning and polishing which can be combined in one process. The EPPo has found its industrial scale applications in treatment of stainless and high carbon steels, nickel, copper, titanium and aluminum alloys. This process has high efficiency, and it meets modern environmental requirements, but its drawbacks include process non-linearity and high power consumption.

To investigate into the process mechanism and to overcome its drawbacks, a novel acoustic emission study has been performed, and a diagnostic approach providing estimation of the surface roughness during the treatment has been proposed. A source of the acoustic emission during the EPPo is a vapor gaseous envelope (VGE) boiling around the workpiece connected as an anode. The EPPo is usually carried out at high voltages in the range from 200 to 500 V applied between the anode and cathode. These conditions promote an appearance of the VGE with a glow discharge intensively modifying the surface. The experimental study has been dedicated to the EPPo of stainless steel BS420S29. The acoustic emission oscillations were measured by a waterproof piezoelectric hydrophone which was *immersed* into the electrolyte and located 6…12 cm away from the workpiece. A data acquisition system consisted of a computer and an analog-to-digital converter, which were controlled by a Labview based program. It was shown that an informative frequency range from 500 to 2000 Hz has characteristic spectral features which help to estimate the surface roughness during the process.

Finally, a new method of the surface roughness diagnostics was designed and introduced into automated EPPo equipment, increasing the process efficiency by 5-7%

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Room Sunrise - Session G5

Atmospheric Plasma Applications

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

8:00am **G5-1 Radiofrequency Cold Plasma Jets Generated at Atmospheric Pressure: from Principles to Applications**, ***Gheorghe Dinescu***, *E Ionita, M Ionita, M Teodorescu, V Marascu, A Lazea-Stoyanova,* National Institute for Lasers, Plasma and Radiation Physics, Romania **INVITED**

We present the principles and various discharge configurations for producing cold radiofrequency plasmas at atmospheric pressure, like plasma jet sources of DBD (Dielectric Barrier Discharges) and DBE (Discharges with Bare Electrodes) types and their utilization in engineering, biomedicine, environment, and nanotechnology. The applicative potential of those plasmas is exemplified with: polymer surface modification in order to control the wettability, carbon cleaning and silicon etching, patterning the cells growth on surface, promoting the adhesion of dental prostheses, operation in liquid phase for chemical decomposition and functionalization of nanomaterials, and synthesis of metallic nanoparticles.

Surface Engineering - Applied Research and Industrial Applications

Room Sunrise - 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, Ali Khatibi, Oerlikon Balzers, Oerlikon Surface Solutions AG

1:30pm **G3-1 Degradation Mechanisms of Protective Coatings in Precision Glass Molding***, **Marcel Friedrichs**, O Dambon, F Klocke,* Fraunhofer Institute for Production Technology, Germany

Thermo-chemical and thermo-mechanical loads act on molding tools during several hundred cycles of Precision Glass Molding (PGM). Wear protective coatings are used on the molding tools in order to protect the optical surface of the molds against degradation. Therefore, the lifetime of molding tools and thus the process efficiency increase. Precious metal coatings such as platinum-iridium (PtIr) are the most versatile material class used for molding various glass types. Furthermore, diamond-like carbon (DLC) and ceramic coatings are employed in PGM as well.

The presented work investigates the degradation mechanisms of different protective coatings by PGM tests at a service lifetime test bench. Subsequent analyses of coated specimens were performed by white light interferometry, scanning electron microscopy (SEM) and X-ray photoelectron spectroscopy (XPS), which proved different degradation mechanisms as diffusion, oxidation, corrosion, glass adhesion and flaking off. Summarizing these observations, a degradation model of the leading degradation mechanisms for different coating systems has been created, which is currently available for further coating development.

1:50pm **G3-2 Nanolayered Coatings for Advanced Fine Blanking Applications***, **Marcus Morstein**, T Schär,* Platit Ag, Switzerland*; B Torp,* PLATIT, Inc., USA, Switzerland*; T Klünsner,* Materials Center Leoben Forschung GmbH (MCL), Austria

A combination of nano- and multilayer structures has proven to provide an optimum combination of wear- and mechanical impact resistance for physical vapor deposition (PVD) coatings used in metal cutting. One field of application where such ceramic coatings are particularly challenged is fine blanking, a versatile metal sheet cutting process able to produce high-quality parts for automotive and general engineering applications, in large quantities. In this process, high compressive and tensile shear forces interact with the coated tool surface, which is additionally challenged by mechanical shock, abrasion and work piece material adhesion.

This paper addresses how using structural design on the nano- and microscale, coatings based on AlCrN and AlCrTiN can be tailored to match the required compromise between wear resistance and toughness. The investigated coatings were produced on two different industrial coating units using lateral (LARC) and central (CERC) cylindrical rotating arc cathodes technology, or a combination of LARC and planar arc cathodes. The nanolayer structure was varied through selection of different target material combinations and different arc currents, and properties relevant to coating adhesion and toughness were measured. In particular, depth resolved internal stress profiles were collected using a modified side inclination XRD technique.

Since wire electrical discharge machining (EDM) is the typical production process for fine blanking punches and dies, pre-treatment methods for substrates made both from powder-metallurgical tool steel and from cemented carbide (WC/Co) were optimized in order to remove surface damage brought in by the manufacturing process and to ensure optimum coating adhesion. Furthermore, a strong coating post-treatment was applied and the beneficial effect of surface smoothness on suppressing workpiece material sticking was illustrated both on the lab scale and in practical fine blanking tests.

Long-term tests in production environment showed that by using the new nanolayered, toughness optimized coatings, controlled wear and thus higher productivity and process stability in fine blanking can be achieved.

2:10pm **G3-3 Growth of Low-defect-density $Ti_{1-x}Al_xN$ Thin Films by Cathodic Arc Evaporation under Industrial Conditions***, **Marta Saraiva**, L Johnson,* Sandvik Coromant R&D, Sweden **INVITED**

The metal cutting industry is, nowadays, an extremely competitive market with numerous significant players. In order to stand out and be ahead of the competition, one needs to be proactive and offer products and solutions to customers before their need for them arises. Such achievement requires to carefully listening the market and continuous strive for improvement. Thus, it is crucial to maintain an R&D activity at the upmost level.

The majority of cemented carbide tools used today are coated, with roughly 50% using a Physical Vapour Deposition (PVD) technique. The ability to deliver world class thin films to our products emanates from possessing adequate equipment and knowledge to tweak the process in order to obtain the desired thin film properties, which result in the best product performance for a specific application. Therefore, the control of microstructure is of high importance to tailor the functional performance characteristics of thin films, and in particular, for hard wear-resistant coatings such as TiAlN. Normally, the grain size of TiAlN is strongly correlated to the Al content, with Al as a grain refiner. Therefore it is of high interest to control the microstructure independently of the Al content. One such example is the recent work by Grezynski *et al.* [1], who demonstrated the growth of low-defect TiAlN by a hybrid DC/HiPIMS magnetron sputtering technique using synchronized bias pulsing under laboratory conditions. Here, we report the discovery of growth conditions for low-defect $Ti_{1-x}Al_xN$, deposited by cathodic arc evaporation using a full-scale industrial deposition system under production-ready conditions. By tuning the process parameters, it was possible to change the ordinary cathodic arc hard coating growth mode of defect-rich grains with featureless surfaces, to a mode of feature rich surfaces and low-defect-density.

[1] G. Greczynski *et al.,* Surf. Coat. Technol. 257 (2014) 15.

2:50pm **G3-5 A Contribution to Explain the Mechanisms of Adhesive Wear in the Plastics Processing by the Example of Polycarbonate***, K Bobzin, T Brögelmann,* Surface Engineering Institute - RWTH Aachen University, Germany*; G Grundmeier, T de los Arcos, M Wiesing,* University Paderborn, Germany*; **NathanChristopher Kruppe**,* Surface Engineering Institute - RWTH Aachen University, Germany

In plastics industry, adhesive wear due to flowing hot melt is one of the main damage mechanisms of extrusion tools. Such damages strongly affect the economic efficiency and the product quality. Due to their beneficial properties, Cr-based nitride hard coatings deposited by physical vapor deposition (PVD) are applied as protective coatings. These coatings can prevent the formation of ferric oxides Fe_xO_y, which influence the adhesion and thus the degradation of plastic melt on the tool surface. In the present work, four different CrAl-based nitride and oxy-nitride monolayer coatings were synthesized on tool steel substrate AISI 420 (X42Cr13, 1.2083) by means of a hybrid direct current and high power pulsed magnetron sputtering (dcMS/HPPMS) process. At this, the chemical composition of the nitride and oxy-nitride coatings was varied in terms of the metal ratio Cr/Al. All coatings and one uncoated steel substrate were analyzed before and after long-term annealing in order to investigate it's influence on the chemical composition of the native passive film at the surface by using X-ray photoelectron spectroscopy (XPS). Furthermore, the influence on the wetting behavior of polycarbonate melt by means of high temperature contact angle measurements as well as on the degradation behavior of polycarbonate by fluorescence measurements using Raman spectroscopy were studied. It was shown that the annealing process leads to a significant higher increase of polycarbonate wetting on the uncoated steel compared to the coated samples. Additionally, the coatings with an increased Al content exihibit a significant lower wetting compared to the other coatings and the uncoated steel. The influence of the metal ratio Cr/Al within the nitride and oxy-nitride coatings on the degradation of polycarbonate was quantified and correlated to the high temperature contact angle measurements.

3:10pm **G3-6 Enhanced Replication Ratio of Injection Molded Plastics Parts by using an Innovative Combination of Laser-Structuring and PVD Coating**, *K Bobzin,* Surface Engineering Institute - RWTH Aachen University, Germany*; C Hopmann,* Institute of Plastics Processing, RWTH Aachen University, Germany*; A Gillner,* Chair for Laser Technology, Aachen, Germany*; T Brögelmann, N Kruppe,* ***Mona Naderi,*** Surface Engineering Institute - RWTH Aachen University, Germany*; M Orth,* Institute of Plastics Processing, RWTH Aachen University, Germany*; M Steger,* Chair for Laser Technology, Aachen, Germany

One of the fast-growing segments of manufacturing is plastics processing. The properties of plastics products can be optimized by a suitable design of the component surface. One promising method is the usage of molding tools structured in the micrometer range for plastics processing by extrusion and injection molding. Such microstructured, optically functional plastics parts are commonly used in light-field photography, displays and security technology. However, the production of optical functional surfaces demands a high quality of replication from the tool insert. Due to the high density of structures and therefore increased surface area the filling rate during the injection molding is very challenging. The adhesion of the plastics melt on the mold surface during the processing can influence the product quality. One possible approach is the combination of physical vapor deposition (PVD) technology with laser based variothermal injection molding to improve the replication of microstructures. PVD hard coatings, such as ternary chromium based nitride (Cr,Al)N are used as protective coating due to the mechanical, chemical and tribological properties to reduce wear and wetting between mold and plastics. Within the scope of this paper, a laser microstructuring was carried out on an injection mold out of AISI 420. A nitride hard coating was deposited on microstructured mold by means of middle frequency pulsed magnetron sputtering (mfMS). Variothermal injection molding with an external laser beam was used to mold microstructured, coated and uncoated molds. The coating morphology and its chemical composition as well as the mechanical and tribological properties were characterized. Commercial plastics polycarbonate (PC) and two types of polymethyl methacrylate (PMMA) were considered for tribological investigations. Adhesion behavior of molten PC and PMMA on (Cr,Al)N hard coatings was analyzed by means of high temperature contact angle measurements. Wear tests were performed by using pin-on-disc-tribometer measurements at room temperature T = 23 °C and at half of the processing temperature T = 110 °C and T = 150 °C against solid PC and PMMA. The results exhibit a high potential of the investigated nitride coating to be used as protective coating against abrasive and adhesive wear during processing of polycarbonate and polymethyl methacrylate. A prosperous replication of microstructured and coated mold could be proofed at different molding temperatures. The replication ratio of optical microstructures is increased significantly up to 20-30 % by using mold coating in comparison to uncoated mold.

3:30pm **G3-7 Sophisticated Wear Resistant Coatings used in Cold Sheet Metal Forming of AHSS Sheet Metals**, ***Ali Khatibi,*** *M Arndt,* Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein

Engineering parts made out of high-strength-steel (HSS) sheets are widely used in the structural reinforcement of the car bodies especially where a high impact and crash resistance is required (e.g., passenger compartment, parts of side doors and bumpers). The increasing need for HSS parts in a typical car body (~40 % in 2007 to 75 % in 2015) on the one hand and development of advanced steel grades with very high strengths (>1000 MPa in UHSS) on the other hand demand the utilization of coatings which in combination with state-of-the-art surface treatments (eg nitriding) provide considerable resistance against a variety of failure mechanisms like adhesive and abrasive wear, galling, chipping, and spallation under severe forming loads.

Nitride based coatings like TiN, TiAlN, TiCrN, and AlCrN, which are also used in cutting tool applications have significantly increased the performance of these forming tool applications when compared to non-coated ones. But as the specific requirements in terms of resistance to abrasive and adhesive wear, fatigue, and in some cases corrosion are completely different for metal forming , there is an increasing demand for dedicated and in many cases complex coatings for an additional gain in efficiency.

The present work addresses the development of the wear and fatigue resistant coatings made by physical vapor deposition (PVD) technique to be used for high-scale industrial forming operations of advanced high strength steel (AHSS) and high strength low alloy (HSLA) sheets. Examples of successful implementation of the coatings in some highly challenging industrial forming applications are presented.

3:50pm **G3-8 Performance Evaluation of HSS Cutting Tool Coated with Hafnium and Vanadium Nitride Multilayers, by Temperature Measurement and Surface Inspection, on Machining AISI 1020 Steel**, ***John H. Navarro-Devia,*** *W Aperador,* Universidad Militar Nueva Granada, Colombia*; C Amaya,* CDT- ASTIN SENA, Colombia*; J Caicedo,* Universidad del Valle, Colombia

The application of hard coatings onto cutting tools improves lifetime, performance and also quality of workpiece, usually by increasing the wear resistant. Hafnium nitride and Vanadium nitride multilayer coating [HfN/VN]n has mechanical, tribological and physic-chemical properties that have been identified by other authors and are desirable for cutting tools. For the above physical vapor deposition (PVD) of hafnium nitride/vanadium nitride [HfN/VN]n multilayer coating, with 1, 50 and 80 bilayers, were carried out onto High-speed steel (HSS) cutting tools by the Multi-target Magnetron Sputtering technique , using toolbit ASSAB 17 3/8 X 3" as a substrate. Toolbits uncoated and coated with HfN/VN multilayers were used to machine AISI 1020 steel samples at the same turning parameters in a CNC machine.

Cutting efficiency, quality product, and tool wear are influenced by temperature, therefore as evaluation method the temperature of the tool, the steel and the chip were measured by means an infrared sensor, a data acquisition system and data analysis in MatLab to identify mean temperature and temperature rate for each tool. Also superficial roughness (*Ra*) of work pieces were evaluated using a roughness tester and Scanning Electron Microscopy (SEM). Tool rake wear were checked through Optical Microscopy and SEM.

In most of the parameters evaluated differences between the tools were identified, as the temperature at the chip increased, temperature at the tool and temperature rates decreased, the work piece roughness reduced up to 25%, and the wear reduces up to 50%, those were proportional to the bilayers number. Results reveals that on [HfN/VN]n coated tools, occurs less deterioration, due the proportionality between the energy transfer and wear resistance, also improves surface finish of the machined piece; all of them are reflected in changes in process temperatures.

Highlights: This novel method relates three fundamental aspects such as temperature of the components, work piece roughness and tool wear, in order to evaluate the performance of coated cutting tools at in situ tests.

The use of multilayer [HfN/VN]n coating on HSS cutting tools, improves superficial properties by reducing friction coefficient and heat transfer, could increase their lifetime, improve the quality of the workpiece, leading to reduce process time and cost, enhance uniformity of material removal and tool lifetime, getting a manufactured product with a better surface quality.

Acknowledgment: This work was supported by funds for internal calls for projects at Universidad Militar Nueva Granada, contract number ING-2100.

4:10pm **G3-9 High Temperature Oxidation and Cutting Performance of AlCrN, TiVN and Multulayered AlCrN/TiVN Hard Coatings**, ***Shi-Yao Weng,*** *Y Chang,* National Formosa University, Taiwan

Transition metal nitrides, such as TiVN and AlCrN, have been used as protective hard coatings due to their excellent tribological properties. In this study, nanostructured AlCrN/TiVN multilayered coatings were deposited periodically by cathodic-arc evaporation (CAE). The AlCrN/TiVN multilayered coatings were post-treated by rapid thermal annealing (RTA). During the coating process of AlCrN/TiVN, TiN was deposited as an interlayer to enhance adhesion strength between the coatings and substrates. The cathode current of both TiV and AlCr alloy cathodes was controlled to produce hard nitride coatings. The microstructure of the thin films was characterized by using a field emission scanning electron microscope (FE-SEM) and transmission electron microscope (TEM), equipped with an energy-dispersive x-ray analysis spectrometer (EDS). Glancing angle X-ray diffraction (XRD) was used to characterize the microstructure and phase identification of the films. The chemical composition and bonding structures were also evaluated. The periodic thickness and alloy content of the deposited coating were correlated with the evaporation rate of cathode materials. A ball-on-disc wear test at room temperature was conducted to evaluate the tribological properties and lubricities of the deposited coatings. To evaluate the cutting performance, end milling tests of 7000 series Al alloys of the deposited tools were conducted at high rotational speed. The TiVN coated tool showed improved tool life as compared to CrAlN and AlCrN because the lubrication effect of TiVN. The design of multilayered AlCrN/TiVN hard coatings is anticipated to inhibit grain growth and improve toughness which expected

to increase the mechanical, tribological and oxidation resistance performances of the coatings.

Surface Engineering - Applied Research and Industrial Applications

Room Grand Exhibit Hall - Session GP

Symposium G Poster Session

GP-2 Oxidation Resistance of Cr_2N and Cr_2WN Coatings Deposited on Ferritic Stainless Steel, *S Yang,* ***Yung-Ting Huang,*** National University of Kaohsiung, Taiwan*; Y Chang,* National Formosa University, Taiwan*; D Lin,* National University of Kaohsiung, Taiwan

Bipolar plate is a key component of solid oxide electrolysis cells, which increases the power density and decreases the costs of the stacks. In general, high operating temperature cause a severe oxidation to decline electrical conductivity. Although Crofer 22 APU (ferritic stainless steel) was developed specifically for SOFC interconnect applications, the oxidation rate is not sufficiently low to enable uncoated Crofer22 APU interconnects to meet the current 40,000 h SOFC lifetime requirement. Therefore, Crofer 22 APU also requires a protective coating both to retard the oxidation rate and to prevent the volatile chromium species.

In this study, Cr_2N and Cr_2WN coatings were selected as the protective coatings deposited on Crofer 22 APU ferritic stainless steel by cathodic arc evaporation. Oxidation kinetics of the Cr_2N- and Cr_2WN-coated samples was evaluated via isothermal tests in atmospheric furnace at 800°C for 1,000 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. The results showed Cr_2N and Cr_2WN-coatings on Crofer 22 APU possessed denser structure and excellent adhesion between coating/alloy. The top scale of Cr_2N and Cr_2WN-coatings belong to spinel structure, but Cr_2WN-coating had higher manganese content than Cr_2N-coating after 1,000 h oxidation. In addition, Cr_2WN-coating possessed smooth surface of scale due to W-doping to decrease surface roughness. After 1,000 h oxidation, both Cr_2N and Cr_2WN-coatings had bilayer scales were Cr_2O_3 and $(Mn,Cr)_3O_4$. Both Cr_2N and Cr_2WN-coatings had excellent anti-oxidation performance; moreover, Cr_2WN-coating had the lower oxidation rate constant than Cr_2N-coating.

GP-4 Synergetic Effect Improved Deposition of Titanium Nitride Films, *C Chang,* Ming Chi University of Technology, Taiwan*; C Ho,* MingDao University, Taiwan*; P Chen,* Da-Yeh University, Taiwan*; W Chen, D Wang,* MingDao University, Taiwan*;* ***Wan-Yu Wu,*** Da-Yeh University, Taiwan

Cathodic arc deposition (CAD) has been widely used in industry for high quality thin film coatings. However, the CAD also produces macro particles or droplets during the deposition process. leading to the degradation of the film properties. Lately, a newly developed physical vapor deposition process known as high power magnetron sputtering (HiPIMS) was found to have the capability of yielding highly ionized flux of both gas and sputtered materials by applying a high power in short pulses to the target. As a result, a smoother and denser thin film with better adhesion to the substrate can be obtained, leading to enhanced mechanical, electrical, and optical properties. However, it was also found the deposition rate of the HiPIMS process was much slower than conventional dc magnetron sputter deposition and CAD. Therefore, a hybrid deposition system combining CAD and HiPIMS was studied in this paper to gain synergetic effect. The two processes were used alternatively in different sequences. Titanium nitride (TiN) film was deposited in such hybrid system to investigate their microstructure and the mechanical properties, including surface roughness, hardness and friction coefficient. We demonstrate that the hybrid deposition system provides a synergetic effect of combining the advantages and compensates the diadvantages of these two deposition techniques. It was found that the macro particles are suppressed. Enhanced hardness was observed in CAD based TiN thin film.

GP-5 Fuel Cell Hot Runner-layer Composite Carbon Bipolar Plates, *S Wu,* MingDao University, Taiwan*;* ***Ai-Huei Chiou,*** National Formosa University, Taiwan*; Y Huang,* Fujian University Of Technology, China

Bipolar plate is one of the important components of proton exchange membrane fuel cell (PEMFC), which in addition to the fuel and the gasifying agent supplied to the reaction zone may be external, and has removed the product, collect current, and as a mechanical support structure of the cell stack . Weight and cost of the proportion of the bipolar plates accounted for 60% and 30% of the battery. Therefore, by using cheap materials and lightweight design to improve the flow channel, will greatly reduce the weight, size and cost of the battery. Therefore, the present study used a lightweight inexpensive PMMA sheet as a bipolar plate body molding material, the use of hot pressing technology has been formed on the upper layer of the PMMA sheet of conductive graphite carbon powder , made of composite bipolar plate. And by hot pressing temperature, time and pressure control, work out the optimum parameters. At the same time, also made out of composite bipolar plate corrosion resistance, electrical conductivity and surface material graphite adhesion effect analysis.

GP-6 Oxinitride Coatings for Milling Tools, ***Joern Kohlscheen,*** Kennametal GmbH, Germany*; V Derflinger,* Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein

Industrial manufacturing has to adapt to an increasing share of difficult-to-cut materials like stainless steels and nickel based alloys. Well-established nitride coatings like TiAlN have limitations in friction behavior and high temperature stability. To overcome some of these limitations, oxide and oxinitride thin films are becoming more popular in PVD coating research and application. The Al-O system is generally considered as first choice and has been studied intensively. In the present work, a well proven AlTiN base layer was modified by adding one or more AlON and AlCrON layers with a thickness of up to 1 micron. Deposition was done by cathodic arc PVD using a commercial system on carbide samples (WC/Co). Al cathode material was evaporated in a plasma of pure oxygen or an oxygen/nitrogen gas mixture. Basic mechanical properties of the resulting multi-layers were determined by universal hardness testing. Structural analysis was done by SEM and XRD. It will be shown that oxidic top layers have quantifiable effects on friction and wear behavior. AlTiN/AlON multi-layers seem to be more resistant against abrasive wear than mono or bi-layer (nitride/oxide) coatings of comparable thickness. The main effect of the oxidic top layer(s) seems to be reduction of the friction in the cutting process. Milling of mild steel (dry and wet) was performed to test the wear behavior of the coated carbide inserts (ISO P). Thermal cracking at the cutting edge could be significantly reduced when oxide top layers were added.

GP-8 Phase Composition, Microstructure Evolution and Wear Behavior of Ni-Mn-Si Coatings on Copper by Laser Cladding, ***Peilei Zhang,*** *X Liu, H Yan,* Shanghai University of Engineering Science, China

Three Ni–Mn–Si coatings were synthesized on copper plate by laser cladding. There are Mn_5Si_2, Mn_5Si_3, Mn_3Si, Ni_3Si, Ni_2Si, Cu_3Si, Mn_3Ni_2Si and $Mn_6Ni_{16}Si_7$ in three coatings. Ni_2Si and Ni_3Si were found in Coating 1# and 2# and there is not any $MnSi_{1.75-x}$ phase in three coatings. Mn_3Ni_2Si phase was found in Coating 1# and $Mn_6Ni_{16}Si_7$ phase was found in Coating 2# and 3#. Phases in Coating 1# should be MnSi + MnNiSi. $Mn_{15}Ni_{50}Si_{35}$ and $Mn_3Ni_3Si_2$ were found in Coating 2#. $Mn_3Ni_3Si_2$ and Mn_3Ni_2Si were found in Coating 3#. Cu_3Si was found in all three coatings. The highest hardness which is about 1100 HV occurs in the clad layer of Coating 1#. Metal silicates (Ni-Si and Mn-Si) are the major factor in increasing the hardness of coatings. There are mainly Ni-Si metal silicates in Coating 1#, Mn-Si metal silicates in Coating 2# and 3#. Hardness of Ni-Mn-Si coatings depends on Ni-Si phases especially Ni_3Si in laser processing. The average friction coefficient for Coating 1#, Coating 2# and Coating 3# is 0.1964, 0.2393 and 0.2582, respectively. Ni-Si phase plays a more important role than Mn-Si in increasing the hardness and decreasing the friction coefficient of coatings.

GP-9 Assessment of Surface Integrity During Machining of Superduplex Stainless Steel Obtained With Three Different PVD Hard Coatings, ***Edinei Locks Junior,*** Católica SC, Brazil*; P Stolf, M Martins,* Centro Universitário Cátolica de Santa Catarina - CATÓLICA -SC, Brazil*; F Amorim, R Diego Torres,* Pontifícia Universidade Católica do Paraná - PUCPR, Brazil*; J Paiva,* Centro Universitário Católica de Santa Catarina - CATÓLICA -SC, Brazil

The Superduplex stainless steel (SDSS) are biphasic materials compound portion of ferrite and austenite with equal parts approximately as well as high levels of chromium and nickel in its chemical composition. This materials are usually employed as pipeline for offshore oil and gas industry. During the cutting process this material presents the follow combination: (i) It shows a tendency to work hardening the surface reflecting in microstructural modifications and residual stress on surface machined; and (ii) It presents high temperatures in the cutting region, resulting in a reduction of tool life. The goal of this work is to evaluate the surface integrity (work hardening and stress corrosion behavior) of the SDSS

obtained after machining process (finish turning) with cemented carbide tools coated with three different PVD coatings. The cemented carbide inserts were coated by Physical Vapor Deposition (PVD) with $Al_{50}Cr_{50}N/Ti_{95}Si_5N$, $Al_{50}Cr_{50}N$ and $Al_{67}Ti_{33}N$. The responses analyzed were tool wear, microstructural characterization, machined surface and stress corrosion analysis. The results indicate that aluminum-rich ($Al_{67}Ti_{33}N$) coatings improve the wear resistance of inserts in this type of machining as well as reduce the surface hardening levels of machined part, reflecting in a better stress corrosion resistance.

GP-11 Surface and Interface Characterstics of CeO_2 doped Al_2O_3 Coating on Solution Treated and Peak Aged AZ91 Mg Alloy, ***Sanjeet Kumar**, D Kumar, J Jain,* Indian Institute of Technology Delhi, India

Mg alloy, being lightest among the engineering materials, has limited use in automotive, biomedical and structural industries due to poor surface mechanical properties. Protection against wear and corrosion may increase the use of Mg-alloys in various engineering applications. Wear and corrosion being the surface related phenomenon, surface modification by various techniques can be sought for improved performance. This paper investigates the deposition and estimation of surface and interface properties of thermally sprayed alumina based coatings. Effect of CeO_2 doping into Al_2O_3 based coatings is also explored. The elastic modulus and hardness at surface and interface both were measured using nanoindentation. In order to investigate the role of substrate microstructure on coating characteristics, the solution treated and peak aged samples of AZ91 Mg alloy have been used as a substrate material. The coatings were characterized using scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), X-ray diffraction (XRD). The results indicate that peak aged substrate microstructure results in coating with better interfacial properties as compared to solution treated case. The role of ceria doping in manipulating the microstructure is discussed in detail.

GP-14 Vacancies in MONTAN – a Mechanism for Tuning the Hardness–toughness Relationship, ***Fedor F. Klimashin**, CDL-AOS TU Wien, Austria; M Arndt,* Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein*; P Polcik,* Plansee Composite Materials GmbH, Germany*; L Lobmaier, N Koutná,* TU Wien, Austria*; D Holec,* Montanuniversität Leoben, Austria*; P Mayrhofer,* TU Wien, Austria

The cubic-structured nitrides of Mo and Ta exhibit excellent tribo-mechanical and electrical properties and are often used as alloying components for improving e.g. materials resistance against wear and oxidation. The outstanding properties both materials owe to their inherent driving force to form vacancies, which are commonly disregarded for any material.

Fusing Mo–N[1,2] with Ta–N[2] – experimentally and theoretically – we have investigated a novel quasi-binary materials system Mo–N–Ta–N. Its natural preference for point defects inherited from relative binaries can furthermore be significantly influenced e.g. by altering the chemical potentials. When dealing with the point defects, the *ab initio* studies are of a great significance: particularly to distinguish between the structures developing “without” point defects and with Schottky defects is anything but trivial experimental issue. Varying the type and volume density of vacancies we present the evolution of structure and mechanical properties of “MoNTaN” coatings. The insights into the origin of the observed phenomena allow us to tune the hardness–toughness relationship and hence design materials for applications requiring tailor-made properties.

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GP-15 Effects of Solidification Behaviour on the Microstructure, Hardness and Corrosion Resistance Properties of Laser Alloyed Al-Fe-Si Coatings, *E Akinlabi,* ***Olawale Fatoba**, E Makhatha,* University of Johannesburg, South Africa

Aluminum and its alloys have been a successful metal materials used for many applications like commodity roles, automotive and vital structural components in aircrafts. A substantial portion of Al-Fe-Si alloy is also used for manufacturing the packaging foils and sheets for common heat exchanger applications. The present research was aimed at studying the morphology and distribution of the iron-containing intermetallics in the Al-Fe-Si alloy. These Fe-intermetallic compounds influence the material properties during rapid cooling by laser alloying technique and play a crucial role for the material quality. Thus, it is of considerable technological interest to control the morphology and distribution of these phases in order to eliminate the negative effects on microstructure. A 3 kW continuous wave ytterbium laser system (YLS) attached to a KUKA robot which controls the movement of the alloying process was utilized for the fabrication of the coatings at optimum laser parameters. The fabricated coatings were investigated for its hardness and corrosion properties. The corrosion performance was investigated in 0.5M H_2SO_4 and 0.5M NaCl solutions at 30^oC via Open Circuit Potential (OCP) and Linear Polarization techniques. The field emission scanning electron microscope equipped with energy dispersive spectroscopy (SEM/EDS) were used to study the morphology of the fabricated coatings and X-ray diffractometer (XRD) for the identification of the phases present in the coatings . The coatings were free of cracks and pores with homogeneous and refined microstructures. The enhanced hardness and anti-corrosion performances were attributed to metastable intermetallic compounds αAlFeMnSi, $Fe_2Si_2Al_2$, Fe_3SiAl_{12}, Al_5Fe_2, Fe_5Si_3, $Al_4Fe_5Si_6$ formed. Computational model used in this research authenticates reasonably with the experimental results.

Surface Engineering - Applied Research and Industrial Applications

Room Sunrise - Session G1

Advances in Industrial PVD, CVD and PCVD Processes and Equipment

Moderators: Emmanuelle Gothelid, Sandvik Coromant R&D Materials and Processes, Ladislav Bardos, Uppsala University, Sweden

8:00am **G1-1 Industrialized HiPIMS**, ***Siegfried Krassnitzer***, *D Kurapov, M Arndt, W Kalss, H Rudigier,* Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein **INVITED**

High power impulse magnetron sputtering (HIPIMS) offers significant advantages over conventional magnetron sputtering because of a much higher degree of ionized species of the sputtered materials. In principle, for hard coating applications, this allows to combine the advantages of arc evaporation – dense hard coatings with excellent adhesion - with smooth surfaces and low defect densities of magnetron sputtered coatings.

HIPIMS includes high power and current density pulses, applied in a reproducible and accurate way. In a classical HIPIMS process, sputter pulses are generated by applying voltage pulses, at different pulse length, followed by a current pulse response. Nevertheless, due to runaway effects at high power density levels, the current response is not very predictable and depends very much on target age, magnetron type or process parameters like reactive gas pressure. To overcome such problems very short voltage pulses are used and current response is mainly given by the current rise time, keeping the current low before reaching a threshold value. It is clear that the discharge properties are always in a transient-like behavior with respect to deposition rate, gas rarefaction and degree of ionization.

A new, different approach, is to apply power-controlled pulses recently described as S3p –technology.

S3p - Scalable Pulsed Power Plasma- overcomes the problem of discharge instabilities by providing a power pulse to establish voltage and current accordingly to the magnetron characteristics.

Key features of recent hardware and process development efforts enable independent adjustment of pulse power density up to 2kW/cm^2 and pulse length in a wide range from 0.05msec up to 100msec at a near perfect rectangular pulse shape for voltage and current. In this presentation examples of S3p-pulses will be presented and the effect on deposition rate and the stability of a reactive sputtering process will be discussed.

A new coating system family was designed around this promising technology. Characteristic data of a small and medium size coating system, together with performance and property data of coatings, produced with S3p-technology like AlTiN, AlCrN, TiSiN, TiN, TiCN also Al2O3 and DLC will be presented.

8:40am **G1-3 Pure HiPIMS Coatings with 2 µm/hour for Cutting Tool Coatings**, ***Christoph Schiffers***, *T Leyendecker, O Lemmer, W Kölker,* CemeCon AG, Germany

Key feature of this new hardware concept is a deposition rate as high as 2 µm/hour for pure HiPIMS coatings. The paper will present how this equipment does a AlTiN FerroCon® coating in 4 hours 20 mins and a TiAlSiN InoCon® film within 5 hours 20 mins. This data are achieved for 100% HiPIMS mode – not hybrid or mixed set-up – and threefold rotation. An integrated concept of an optimized magnetic set-up of the magnetrons together with the door assembly design of the cathodes – HiPIMS without cable, the pulse unit is right on the chamber door – and a full synchronization between the HiPIMS sources and a dedicated table Bias makes this so far unachieved rate possible.

A scratch load of 120 N for a TiAlSiN coating on a sintered carbide surface indicates enormously high plasma ionization. The dense nature of the films is revealed by nano indentation results showing so far not reachable H³/E² values. SEM images of the fine grain morphology underline this. Machining tests in TiAl6V and in stainless steel show that pure HiPIMS takes the performance of cutting tools to a premium level.

A case study on TiB_2 coatings illustrates the benefit of pure HiPIMS coatings: this technology adds to the advantages of sputtering – smooth, droplet free coatings and an unlimited choice of the chemical composition – a tremendously high ionization and hence best adhesion of a dense and uniform coating. The pure HiPIMS technology broadens the application range of TiB_2 to cutting tools for highly abrasive workpiece materials.

9:00am **G1-4 Deposition of Acrylic Acid on Argon or Air Atmospheric Pressure Plasma Treated Silicon using a Novel Chamber Design**, ***Wei-Yu Chen***, University of Sheffield, UK; *A Matthews,* University of Manchester, UK; *F Jones,* University of Sheffield, UK

Plasma technologies are of great industrial interest due to their non-polluting nature and possibility to provide rapid treatment times. Whilst vacuum plasma processes have received intense attention over recent years, we are now also seeing increased interest in atmospheric plasmas. For example, peroxide groups formed via atmospheric pressure plasma (APP) activation are favorable for free-radical surface graft polymerization of acrylic acid (AAc). AAc grafting on a material offers carboxylic acid functional groups on its surface, which opens possibilities to find wider uses in different applications, such as biosensors, biocompatibility improvement and enhancement of the interface adhesion in composite materials. In this study, APP treatment with different carrier gases, air and argon, and vapour phase grafting were applied in a bespoke Pyrex glass chamber to deposit of AAc on silicon wafers. To limit the effect of atmospheric gas on the process and prevent AAc monomer from fragmenting during APP, a plasma activating zone and a grafting zone were designed and located separately inside the bespoke chamber. Silicon specimens were first activated using air- or argon-APP, then shifted to the grafting zone downstream of the AAc vapour inlet. The surface wettability was evaluated by water contact angle analysis and chemical composition was identified by X-ray Photoelectron Spectroscopy (XPS). Contact angles with water were initially 56.1±0.8° and after APP treatment with air and argon were 10.2±0.8° and 4.6±0.2° respectively, and increased to between 10.0° to 16.0° after AAc vapour grafting. The binding energy peak at 289 eV from XPS also indicated the presence of carboxylic acid groups on the surfaces. A higher intensity of the peak at 289 eV was also detected, compared with the surface treated by APP with AAc vapour injecting to the activating zone during the treatment.

Keywords: Atmospheric pressure plasma treatment, Acrylic acid, Carboxylic acid groups, Surface grafting.

9:20am **G1-5 Reactive Deposition in the Magnetized Hollow Cathode Activated Magnetron**, ***Hana Barankova***, *L Bardos,* Uppsala University, Angstrom Laboratory, Sweden

Recently, a new type of the magnetron, Magnetized Hollow Cathode Activated Magnetron has been developed, where the target is coupled with the hollow cathode magnetized by the magnetic field of the magnetron. This configuration, producing intense and stable plasma in a wide interval of pressures brings about enhanced magnetron performance and increases the deposition rate. The results of the TiN reactive deposition are presented and discussed. Increased deposition rate compared to the metal deposition rate is indicated for TiN. This is consistent with previous results obtained for the hollow cathodes.

9:40am **G1-6 Ionisation Enhancement Control for Magnetron Sputtering Processes**, *V Bellido-Gonzalez, F Meyer, T Sgrilli, H Li,* ***Frank Papa***, Gencoa Ltd., USA; *J Housden, L Espitalier, S Banfield,* Wallwork Cambridge Ltd, UK

Hot Filament technology has been used for a long time for the ionisation enhancement of different PVD technologies such as Electron Beam Evaporation (eBE) and Magnetron Sputtering (MS). These applications already form part of standard industrial production processes in varied coatings such as eBE TiN and Magnetron Sputtered DLC.

In terms of large area scalability, Magnetron Sputtering is one of the most attractive PVD technologies. However, the basic MS process, with no assisted filament, would generally be lacking in ionisation. When a higher degree of ionisation is needed, an additional technology needs to be coupled, such as inductively coupled RF or HIPIMS which offer the possibility of higher plasma density. Both methods however present challenges when it comes to large area. RF discharges become difficult to handle and control over large areas. In a similar way, HIPIMS discharges on large area cathodes present some challenges, such as the levels of peak power delivery ability from commercial power supplies, arcing control, tuning and cabling.

An alternative method to achieve enhanced ionisation for large area systems would be to add a hot filament based technology for enhanced electron emission. Although this method could offer a simpler and more economic route to large area industrialisation, further control of the ionisation enhancement is needed. Design of the deposition system, integration and control of the ionisation enhancement is necessary in order

to achieve the consistency in the results demanded in industrial production.

Steering and confinement of the electrons emitted from the filament are critical for process enhancement.

Improvements in coating properties for AlNOx and AlCrN will be shown as well as the possibilities to "tune" the plasma density.

10:00am **G1-7 Bipolar Sputtering - Waveform Adaptability in Plasma Applications**, ***Wojciech Gajewski,*** *K Ruda, J Swiatnicki, P Ozimek,* TRUMPF Huettinger Sp. z o.o., Poland

New applications and increasing requirements on cost effective processing stimulates the use of innovative solutions for the precise control of the plasma. Increasingly important in determining the optimal plasma parameters are the functionalities of the power supply The flexibility in output current and voltage shape modification, tunable pulsing frequency, advanced arc and power delivery management are the driving factors for successful usage of bipolar technology in a variety of industrial applications. Furthermore, the flexible design enables an easy up-scaling form single kilowatt up to 180 kW in the biggest industrial sputtering systems.

The flexibility and modifiability of bipolar power supplies are the key factors making them an interesting alternative for the classical MF units with sinusoidal output. This contribution summarizes author's many years of industrial experience with the application of bipolar technology to summarize the advantages and challenges associated with its exploitation.

First, the dependence of TiN coating properties on different output frequency will be discussed in details. The discussion will be followed by the impact assessment of the output signal shape on plasma and coating properties. Furthermore, functionalities distinctive for bipolar power supplies will be briefly described in comparison with competitive power delivery technologies. As next, deposition from a ceramic target will be used to discuss the relationship between the available arc detection and suppression algorithms and plasma stability. It will be shown how a combination of novel arc management algorithms together with applicability of different pulsing frequencies can be used for process stabilization by reduction of arcing probability. As a summary, cost and benefit statement of industrial implementation of bipolar technology will be emphasized based on latest process results.

10:20am **G1-8 New Hauzer CARC$^+$ Technology Dedicated to Nitriding, Etching and Coating Process**, *J Zhu, G Negrea, M Eerden, D Doerwald,* ***Roel Tietema,*** IHI Hauzer Techno Coating, Netherlands

CARC$^+$, Hauzer lately developed circular arc technology, marked the birth of a new generation of hard coatings, opening up new dimensions of coating performance in various application fields. Thanks to the advanced source architecture of CARC$^+$ technology, the energy and charge status of the species in the generated plasma can be tuned in a wide range by combined configuration of coil magnets and permanent magnets such that, the most advantageous nitride and carbide hard coatings, controlled by its microstructure and composition, are obtained. In addition, the CARC$^+$ source is also able to act as an anode, obtaining much homogeneous and enhanced nitriding and/or etching effect on the to-be-coated parts.

In this work, we will show the microstructure and cutting performance of TiN and TiCN coated HSS tools, processed with CARC$^+$ anode etching and nitriding. Meanwhile, microstructure evolution and cutting performance of AlCrN (64/36 at.%) coating using various cathode magnetic fields will be addressed.

10:40am **G1-9 Characterization of Advanced Coating Architectures Deposited by the HI3 Process**, ***Joerg Vetter,*** Oerlikon Balzers Coating Germany GmbH, Germany*; K Kubota, M Isaka,* Mitsubishi Hitachi Tool Engineering, Japan*; J Mueller, T Krienke,* Oerlikon Balzers Coating Germany GmbH, Germany*; H Rudigier,* Oerlikon Surface Solutions AG, Liechtenstein

The tailoring of coating architectures of vacuum arc deposited coatings is limited to specific cathode material properties which are evaporable by the arc process. The HIPAC magnetron sputtering process (a classical HiPIMS process) can be used to atomize and ionize materials which are difficult to evaporate or not evaporable by cathodic arc . Both deposition methods are running as individual process steps, but also in a hybrid mode. This HI3 process opens a process window to generate advanced coating architectures. Selected advanced coating architectures are highlighted. Multilayer coatings containing sophisticated nano multilayer structures are presented. Both basic coating characteristics like macroscopic stress, x-ray diffraction and mechanical properties and TEM and SEM investigations. are presented.

11:00am **G1-10 Mechanical Property and Thermal Stability of Multicomponent AlTiSiN and AlTiBN Hard Coatings using Ternary Alloy Arc Sources**, ***Meng-Chun Cai,*** *Y Chang,* National Formosa University, Taiwan

Ternary transition metal nitride, such as TiSiN and TiBN, have been attracting great interest for industrial application as hard protective coatings due to their high hardness, wear resistance, tribological properties, and chemical stability. In this study, AlTiSiN and AlTiBN coatings were synthesized by cathodic-arc evaporation. During the coating process of AlTiSiN and AlTiBN, TiN was deposited as an interlayer to enhance adhesion strength between the coatings and substrates. The AlTiSiN and AlTiBN coatings were annealing by rapid thermal annealing at high temperature. The microstructure of the synthesized 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 coatings. Mechanical properties, such as the hardness and elastic modulus, were measured by means of nanoindention and Vickers hardness measurement. Ball-on-disc wear tests at room temperature and high temperature (500℃) were conducted to evaluate the tribological properties of the coatings. To evaluate the correlation between impact fracture resistance and hardness/elastic modulus ratio of the deposited coatings, an impact test was performed using a cyclic loading device with a tungsten carbide indenter as an impact probe. The AlTiSiN coating is anticipated to increase the hardness and wear resistance, which were expected to increase film thermal stability and abrasion resistance. The AlTiBN coating is anticipated to increase the adhesion, hardness, toughness and tribological resistance.

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