Surface Engineering - Applied Research and Industrial Applications
Room Pacific Salon 1 - Session G1+G3-ThM

Advances in Industrial PVD, CVD, and PECVD Processes and Equipment/Innovative Surface Engineering for Advanced Cutting and Forming Tool Applications

Moderators: **Ladislav Bardos**, Uppsala University, Sweden, **Emmanuelle Göthelid**, Sandvik Machining Solutions, **Ali Khatibi**, Oerlikon Balzers, Oerlikon Surface Solutions AG, **Christoph Schiffers**, CemeCon AG, Germany

8:00am **G1+G3-ThM-1 Predicting Coating Uniformity and Cathode Utilization in Magnetron Sputtering Applications using Numerical Simulation***, **Adam Obrusnik**, P Zikan,* Plasma Solve, Brno, Czech Republic

In many disciplines of applied science, numerical simulation and computer-aided engineering (CAE) are well established and are being commonly used in process design and utilization. This is not the case in plasma processing science, where numerical simulation is still largely considered an academic endeavour. This is partly due to the complexity of the problem and partly due to the lack of tools available.

In this contribution, we aim to illustrate that numerical models of PVD processes and the codes necessary for implementation of these models have matured enough and are ready to be used on the industrial scale. We provide real-life examples as to how a numerical simulation can be instrumental in optimization of magnetron sputtering processes and low-pressure cathodic arc PVD. To that end, we utilize solvers relying both on continuum plasma models and particle-based models (DSMC, direct-simulation Monte Carlo). These solvers are largely based on open-source computational libraries OpenFOAM and Elmer, which allows for excellent computational scalability and computation in realistic 2D and 3D geometries. The codes presented are capable of predicting application-relevant observables, e.g. magnetron cathode consumption, multilayer structure and coating uniformity on 3D rotating samples.

The contribution illustrates the potential of open-source computational solutions as well as cloud computing, which might be necessary for addressing industrial-scale plasma simulations.

8:20am **G1+G3-ThM-2 Multinary HiPIMS***, T Leyendecker, W Koelker, S Bolz, **Christoph Schiffers**,* CemeCon AG, Germany

The almost unlimited choice of materials is one of the biggest strength of HiPIMS. It offers a variety of opportunities for tailoring coatings by alloying of the chemical composition or by using species from certain elements to densify the film by heavy ion bombardment.

Carbon based coatings are mainly used for tribological applications. However, coatings such as TiCN and WC/C are of high relevance for tap tool applications – a market of about 100 Mio tools per year. Commercial coating products for the threading industry are most frequently multilayer designs of an AlTiN with a carbon containing top layer. This gives HiPIMS a new challenge: multinary coatings of materials with rather different properties.

This paper will introduce a new HiPIMS control concept offering for every source an individual set of HiPIMS pulse parameters. Now the coating designer can take the very different ionization potential of different materials into account or select a source for heavy ion bombardment while the other ones are optimized for highest sputtering rate.

All this with full synchronization of the HiPIMS cathodes to the HiPIMS table bias. Hence, the film's designer can actively go for the ionized target species while suppressing the incorporation of sputtering gas into the film.

Data from plasma analysis as well as hardness and stress measurement reveal a huge effect of the pulse parameters such as frequency and pulse length on the film properties.

Multinary HiPIMS gives freedom to coating engineers and broadens the application range of HiPIMS.

Case stories from the thread tool industry underline the industrial relevance of the concept.

8:40am **G1+G3-ThM-3 From Small Parts to Particles – Experiences in Bulk Coating***, **Heidrun Klostermann**, F Fietzke, B Kraetzschmar,* Fraunhofer FEP, Germany

Bulk coating seems to be an intriguing variant of vacuum coating for small mass parts. Compared to individual part coating, the handling effort is considerably reduced. This applies to indirect and direct labor such as the production and maintenance of adapted fixtures and the charging and de-charging of individual parts. Furthermore, the utilization of processing volume can be maximized, avoiding void space between the parts. This benefit grows with decreasing part size. However, when it comes to particles, new challenges arise in bulk coating that thwart the fast success.

The surface to be coated per volume increases with decreasing size of the parts/particles. Therefore, high deposition rates are required in order to keep the processing time reasonably low. The economic assessment of bulk vacuum coating has to be made for every individual coating task including a specific substrate, the intended function of the coating, the production volume and the costs. Certainly, for many high volume bulk goods vacuum coating is not an option. However, novel high tech materials are often composites in which bulk materials are included and combined with metals or polymers to result in improved properties and/or reduced weight. Correspondingly, surface functionalization of bulk material becomes more important and more requested.

Fraunhofer FEP has pursued the objective of vacuum bulk coating for several years. We have developed a technology for corrosion resistance coating of rivets. Based on the barrel coating device ALMA 1000, which includes the high rate plasma activated evaporation technique as well as the magnetron sputtering technique, other bulk materials are envisaged for surface functionalization. The challenge starts with the handling of particulate material, its behavior in the rotating substrate drum and its implication for the vacuum process.

First experiences with different particulate materials will be presented that illustrate options and limitations. The contribution is thought to stimulate new ideas for surface functionalized bulk materials that can eventually be solved by vacuum plasma treatment and coating processes.

9:00am **G1+G3-ThM-4 A Novel Industrial Coating System for the Deposition of Smooth Hard Coatings Combining HiPIMS V+ and Rotatable Magnetrons***, **Herbert Gabriel**, J Santiago Varela,* PVT Plasma und Vakuum Technik GmbH, Germany*; I Fernandez,* N4E Nano4Energy S.L.N.E, Spain*; N Dams,* PVT Plasma und Vakuum Technik GmbH, Germany*; A Wennberg,* N4E Nano4Energy S.L.N.E, Spain*; J Lu,* PVT Harbin Coating Ltd, China

Multi-layered, nano-structured metal-nitride and carbo-nitride coatings are very well established in the cutting tool industry as well as in other industries. For years most of such coatings have been deposited by arc evaporation despite the badmouthed "droplets", since arc evaporation is an extremely economic process with significant advantages such as high intrinsic ionization which is particularly beneficial during metal etching.

Magnetron sputtering with its low ionization and its deficiencies in adhesion and productivity significantly improved with the development of HiPIMS. An even more significant improvement is the HiPIMS V^+ process where adding positive reverse pulses creates enhanced ion assistance and incorporation to the growing film, thus also increasing the deposition rate .

On the other hand, rotatable magnetrons are well known to provide better material usage, longer operation and higher operation power levels.

The novel industrial system introduced in this paper shows the unique combination of HiPIMS V+ with rotatable magnetrons in a batch coater system, thus enhancing system productivity. This process is applied in a multi-cathode magnetron sputtering system using 4 pieces of 1 m long rotatable cathodes equipped with a strong unbalanced magnetic design allowing high ion-to-neutral ratios to the substrate. The system can be configured to operate in unipolar HiPIMS, Dual Bipolar HiPIMS or DC-Pulsed.

Besides a description of the newly designed coating system, nitride and carbo-nitride nano-structured multilayered coating structures based on Ti, AlTi, AlCr and TiSi deposited in such system are shown and characterized, concerning their micro-structure, adhesion, microhardness and composition.

Wear and performance data are presented.

9:20am **G1+G3-ThM-5 From DCMS to HiPIMS: A Giant Leap for Cutting Tools?***, **Bastian Gaedike**,* Hartmetall-Werkzeugfabrik Paul Horn GmbH, Germany **INVITED**

Carbide cutting tools for machining (e.g. milling or turning) are coated with physical (PVD) or chemical vapor deposition (CVD) to meet the high requirements. Arc and DCMS (Direct Current Magnetron Sputtering) have dominated the PVD sector for decades.

In recent years the PVD process HiPIMS (High Power Impulse Magnetron Sputtering) has moved more and more into the focus of the cutting tools industry. Research in this area has already progressed considerably. Since the observation of high peak currents by high-frequency pulsing by Kouznetsov in 1999, researchers have published several hundred publications on HiPIMS. While the technical details of the HiPIMS process are highly interesting for researchers and developers, the focus for users and manufacturers of cutting tools is more on the use of coatings.

The machining of new materials for aerospace and medical technology has greatly increased the demands on cutting materials and cutting edge geometries. In general steel and stainless steel processing, there is also an increase in downtimes and cycle times. For the manufacturers themselves, not only the performance of the layers is decisive, but also the economic efficiency in production. The low deposition rates of HiPIMS, which are often reported in publications, are a major topic here.

The main topic of the presentation is the requirements of users and manufacturers of cutting tools and the resulting challenges for research and development. In addition to various applications and problems in machining, the coating of sharp cutting edges and the problem of the deposition rates of HiPIMS are under discussion.

10:00am **G1+G3-ThM-7 Application of Twin-Roll PECVD for Surface Functionalization on Flexible Substrate***, Y Isomura, Y Ikari,* ***Tadao Okimoto,*** Kobe Steel, Ltd., Japan

Technological development of surface functionalization is of great significance today in the wide field of application. This ranges, for instance, from hard nitride coating for cutting tools to functional transparent electrode on a flexible polymer film. For this purpose, plasma deposition and treatment techniques of cathodic arc, sputtering, Plasma Enhanced CVD (PECVD) are largely used in an industrial production scale. A batch type system is commonly employed in hard coating application where a variety of individual components is deposited by tailor-made coatings while a continuous production with Roll to Roll process, hereafter referred to as R2R, provides a high productivity for surface functionalization on a flexible substrate. Application of R2R has been expanding due to a recent increase in demand for smart devices such as functional display, wearable devices and sensors, energy conversion and storage, contributing towards realization of sustainable smart society in the near future.

We have developed the so-called Twin-Roll PECVD technique integrated into R2R process in an industrial scale. A unique feature of Twin-Roll PECVD is that the winding rolls themselves for flexible substrate serve as the electrode for discharge of PECVD process. This provides a stable discharge for a long time compared to a conventional pulse PECVD since no deposition occurs on the surface of electrode, which often limits productivity of deposition of non-conductive coating such as oxide and nitride. Optimization of magnetic field on the electrode enables to sustain the discharge at a relatively low pressure of 1~10 Pa. The low pressure PECVD process suppresses the formation of dust particles, and hence the film deposition with a low defect density is realized.

In this work we report the basic principle of Twin-Roll PECVD as for a new type of plasma processing as well as some applications of surface functionalization on flexible substrate using this technology. We have deposited and characterized SiOx films exhibiting a high water vapor barrier property, SiOx/SiNx films with a high refractive index, and DLC (Diamond-Like Carbon) films showing a high infrared transmissivity. Spectrophotometry, XPS, TOF-SIM, water vapor transmissivity measurement were mainly used for film characterization. In addition an application of Twin-Roll PECV for deposition of heat-ray reflecting film is demonstrated.

10:20am **G1+G3-ThM-8 A New System Platform for Ultrafast Nitriding and Diamond Like Carbon (DLC) Deposition Based on a Hollow Cathode Discharge***,* ***Frank Papa****, T Casserly, A Tudhope, S Gennaro,* Duralar Technologies, USA

Diamond Like Carbon (DLC) has become one of the most important coatings in the Physical Vapor Deposition (PVD) industry due to its chemical inertness, hardness and low coefficient of friction. For mass production, these coatings are usually produced in large batch coaters with PVD interlayers for adhesion and load absorption with a Plasma Enhanced Chemical Vapor Deposition (PECVD) DLC layer on top. Typical cycle times for such a batch system are on the order of 6-9 hours. A new PECVD system platform based on hollow cathode technology has been developed for the deposition of DLC (a-C:H) with a complete door to door cycle time of less than 30 minutes for a 2-3 µm DLC coating. In addition to high rate DLC coating (0.5 to 1 µm/minute), ultrafast plasma nitriding can also be done before the DLC process in order to harden and chemically modify the surface before DLC coating. Titanium alloys, stainless, carbon and alloy steels are suitable materials for such processes. In addition to the short cycle times, three dimensional parts with aspect ratios of 15:1 (length:diameter) can also be coated with coating on both external and internal surfaces. DLC coating thicknesses greater than 50 µm can be achieved.

10:40am **G1+G3-ThM-9 Combinatorial Development of Nitride and Oxide Thin Films on an Industrial Scale***,* ***Rainer Cremer,*** KCS Europe GmbH, Germany **INVITED**

The ever increasing complexity of modern coatings triggers the need of sophisticated technologies for rapid and commercially advantageous development methods. One possibility to significantly increase the speed of materials development is the use of combinatorial approaches.

In this paper, the applicability of such combinatorial methods in industrial development of advanced materials is illustrated presenting various examples for the deposition and characterization of one- and two-dimensionally laterally graded coatings. These coatings were deposited by means of magnetron sputtering, arc ion plating and plasma-enhanced chemical vapor deposition.

To illustrate the advantages of this approach for the industrial development of advanced materials, the multi-component metastable hard coatings (Ti,Al)N, (Ti,Al,Cr)N and (Ti,Al,Si)N as well as various non conductive oxides and nitrides were investigated with respect to the relations between structure and composition on one hand and physical properties like hardness, erosion resistance, cutting performance and oxidation behavior of these coatings on the other.

11:20am **G1+G3-ThM-11 Protective, Tribological and Decorative PECVD Coatings Deposited with a New Microwave Source: Plasma and Layer Characterization for Appropriate Applications***,* ***Rolf Schäfer****, T Radny, K Nauenburg,* robeko GmbH & Co.KG, Germany*; S Ulrich,* Karlsruhe Institute of Technology (KIT), IAM, Germany

The KIT designed a novel MW source with a parabolic reflector to focus the high microwave plasma density directly onto the substrates during the deposition of scratch, corrosion and wear protective, tribological, biocompatible and decorative coatings by a PECVD processes using e.g. siloxane and hydrocarbons as precursor compounds. A number of MW sources can be arranged in line or even lateral to form a larger coating area to be used in Inline coaters or larger batch coating systems, also being easily combined with other PVD sources e.g. planar and rotatable sputter magnetrons, both with the option to be driven with a HiPIMS generator to create new and promising combination of diverse plasma effects. At the very first it is necessary to characterize the plasma by different diagnostic methods, e.g. Langmuir probe measurements, OES, RFA and others, but also work out the influence of the plasma parameter settings for the properties of the deposited layers, e.g. diamond - or quartz-like-coatings. At least the design has to be optimized in details to ensure a long-lasting reliable stability of process conditions and product properties in industrial production. First results for all issues will be presented.

11:40am **G1+G3-ThM-12 Complex Coating Technique for Smallest Part of Advanced Powertrain Fuel System***,* ***Sung Chul Cha****, H Park, J Lee,* Hyundai Motor Group-Hyundai Kefico, Republic of Korea*; K Ko, C Shin,* Dongwoo HST Co. Ltd., Republic of Korea

The objective of this paper is to achieve the high quality SiO-DLC (Diamond Like Carbon) coating on small spherical part with diameter of 2-4 mm applied to the advanced powertrain fuel system. Silicon and oxygen incorporated DLC reduces internal residual stress and improves high temperature stability compared to DLC. The spherical part moves continuously up and down and hits their counterpart. Therefore this part is required to have high hardness and wear resistance. Conventional process, this part firstly welded with bar shaped part and then assembled with further parts, finally coated as assembled state. However in this manner, the maximum amount of charging in one coating batch of coating machine is limited due to big size of whole component, causing cost increasing. Furthermore the assembled component can be contaminated by alkali cleaning agent during cleaning process before and after coating and discolored by the process gas during coating process. Therefore the spherical part is to be coated before welding on the bar shaped part.

One of the challenges of this work was mounting the spherical parts in the coating jig. To maximize the production amount, parts shall be mounted in vertical direction in the coating machine. Therefore the appropriate jig is designed and installed with a magnet substrate backside of the jig to magnetically hold parts. The other challenge was designing for masking in the jig. At least 0.7 mm height of non-coating region must be secured concerning heat damage on coating by laser welding process. Thus the parts should be mounted on the masking jig with high precision. The tolerance of the jig had to be as small as possible to block the inflow of coating material into the gap between spherical part and jig. Moreover the jig should be as thin and narrow as possible to maximize the production amount.

The coating is composed of three layers, Cr as bonding layer, WC or CrN as buffer layer and SiO-DLC as functional top layer. Bonding and buffer layers are coated with reactive or non-reactive sputtering method and top SiO-DLC layer is coated by reactive sputtering with HMDSO (Hexamethyldisiloxane). The minimum properties of hardness was 20 GPa, of coating thickness 1.5-2.5 um and of roughness lower than Ra 0.05 um. With only PACVD, coating hardness of part's equator zone could not be satisfied due to the limitation of complex shape. In conclusion, SiO-DLC coating technique by reactive sputtering with high precise jigging and masking technique resulted best properties for modern powertrain fuel system, detailed described in this paper.

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