Surface Engineering - Applied Research and Industrial Applications
Room Sunset - Session G1

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

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

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

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

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

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

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

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

The exceptionally hard diamond layer provides the perfect foundation to the HiPIMS film. Diamond has an outstanding thermal conductivity and spreads the extreme heat that is generated in the cutting zone. Both effects, the unrivalled hardness and the heat spreading properties of diamond, avoid the typical egg-shell effect. The generally lower heat input into the carbide substrate and the avoidance of local overheating is an enormous plus when it comes to cutting of heat resistant superalloys.

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

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

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

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

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

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

The technology is very much up-scalable.

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

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

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

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

HiPIMS differs significantly from conventional d.c. magnetron sputtering, and together they cover a wide range of scenarios of deposition kinetics that are often critical for the characteristics of resulting coatings. In d.c. magnetron sputtering, particles sputtered from the target are mostly electrically neutral with low kinetic energies of only a few eV. In HiPIMS deposition, however, the particles sputtered from the target crossing the highly densed plasma are largely ionized and, therefore, their kinetic energy can be purposefully adjusted before arriving at the deposition surface via an appropriate substrate bias. Additionally a novel microwave plasma source designed for large-area industrial plasma surface processing and film deposition creates high density plasmas at low pressures even far from the source. A hybrid deposition method (HiPIMS, d.c. magnetron sputtering and microwave plasma source deposition) is developed for advanced carbon-based nanocomposites in the system Ti-Zr-C-N. The coating properties and microstructures were characterized and compared. The results will be discussed in this study and related to the strongly different deposition kinetics. The coatings were deposited from a segmented metallic Ti/Zr target in reactive mode with different Ar-N_2-CH_4

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

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

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

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

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

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

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

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

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

Graphene and graphene-like-carbon films have found broad applications ranging from corrosion protection of metals to transparent conducting electrodes. Here, we study a high-throughput, roll-to-roll plasma chemical vapor deposition (CVD) of graphene-like-carbon film on Cu foil, particularly the related effects of heat transfer and web speed on film quality in combination with optical emission spectroscopy (OES) to study plasma composition during growth. Graphene-like carbon film is deposited on a moving copper foil using a custom-built roll-to-roll radio-frequency plasma CVD system. The carbon film is deposited on both sides of the substrate at a high speed of 300 mm/min. We find an asymmetry in the quality of the film on the two sides of Cu foil attributed to asymmetry in the plasma's sheath. Results from OES provide insights into the distribution of chemical species between the electrodes. Emission intensities from major plasma species (i.e., CH, C_2, H and H_2) show no dependence on web speed. However, the temperature of the Cu foil, estimated from its blackbody emission using OES, decreases with increased web speed. The Cu foil temperature is near 1000 K for plasma power of 1350 W. We also find that high web speed is necessary with high-energy plasma input to achieve an optimal dwell time and minimal defects in graphene-like carbon film. The results provide insights into roll-to-roll growth of graphene-like carbon film on Cu foil using heat transfer principles and establish the feasibility of the method for a large-scale production.

11:20am **G1-11 TAOS Based Cu/TiW/IGZO/Al_2O_3/Pt Bilayer CBRAM for Low-power Display Technology***, **Kai-Jhih Gan**, P Liu, W Chang, D Ruan, T Chien, Y Chiu, S Sze,* National Chiao Tung University, Taiwan

Herein we report a Cu/TiW/IGZO/Al_2O_3/Pt bilayer CBRAM stack using amorphous InGaZnO, a TAOS material, as the resistive switching layer. The addition of a thin metal-oxide layer (5 nm-thick Al_2O_3) in the bottom of the IGZO memory stack significantly increases the R_{OFF} and the memory window. The a-IGZO bilayer CBRAM shows the excellent memory performances, such as low operation current (down to 10μA), high on/off resistance ratio (more than 10^3), high switching endurance (up to 10^3 cycles)and the capability of multi-level tuning have been demonstrated in our memory device. Meanwhile, high thermal stability was also achieved (two decades of window margin are constantly maintained beyond 10^4 s at 85 °C). This result has given a great potential for the TAOS based material utilizing in CBRAM stacks and integrating into the display circuit for future memory in pixel application.

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