Friday Morning, April 28, 2017

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 H^3/E^2 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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