Hard Coatings and Vapor Deposition Technologies
Room Golden West - Session B1-2

PVD Coatings and Technologies

Moderators: Joerg Vetter, Oerlikon Balzers Coating Germany GmbH, Qi Yang, National Research Council of Canada, Jyh-Ming Ting, National Cheng Kung University

2:10pm **B1-2-3 Impact Analysis of Power Source Operating Parameters on Hardness, Adhesion and Film Composition of TiN Functional Coatings**, *K Ruda, W Gajewski,* ***Jakub Świątnicki****, A Oniszczuk,* TRUMPF Huettinger Sp. z o.o., Poland

ncreasingly important in determining the optimal plasma processing parameters are the functionalities of the power supply. The flexibility in output current and voltage shaping, 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. 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 recent industrial experience with the application of bipolar technology for deposition of protective and decorative TiN coatings.

First, the dependence of the deposition rate on different output frequency will be discussed in details including the relationship with the process stability due to arcing in reactive processes. 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. The discussion will be followed by the impact assessment of the output signal shape and frequency on TiN film hardness. Films prepared with different power delivery settings show comparable adhesion property with critical load in the range of 62 N, hardness *H* > 6500 Mpa and Young's modulus *E* ~ 300 Gpa. The distinct differences in the film properties obtained with different operating parameters will be discussed in details. The discussion will be summarized with cost and benefit analysis of industrial implementation of bipolar technology based on process results.

2:30pm **B1-2-4 Substitution of Commercially Coated Tungsten Carbide Tools in Dry Cylindrical Turning Process by HiPIMS Coated Niobium Carbide Cutting Inserts**, *E Uhlmann,* ***Daniel Hinzmann****, K Kropidlowski,* Institute for Machine Tools and Factory Management - Technical University Berlin, Germany; *P Meier,* Institute for Machine Tools and Factory Management - Technical University Berlin; *L Prasol,* Institute for Machine Tools and Factory Management - Technical University Berlin, Germany; *M Woydt,* BAM Berlin, Germany

Coated tungsten carbide (WC) tools are applied for machining of various workpiece materials in industrial applications. The recent development of alternative cutting materials shows the potential of niobium carbide (NbC) for turning of iron-based materials. Its use in cutting tool applications is based on advantages regarding hot hardness and low solubility of binderless NbC in solid chrome, nickel, cobalt or iron. With a reduced tendency for adhesive and diffusion wear in uncoated state, a higher material removal rate combined with a higher process reliability at increased cutting speed represents state of the art machining results. In order to draw a comparison between commercially available WC tools, NbC tools are tested in uncoated and coated condition during dry cylindrical turning of carbon steel C45E. Two different NbC substrate materials are included in machining trials, each differing in chemical composition and mechanical properties. A cobalt (Co) bonded niobium carbide defined as $NbC_{0.88}$-14Co and a nickel (Ni) bonded NbC with the specification $NbC_{1.0}$–12Ni4Mo4VC are selected and compared to submicron grain WC-6Co tool material. The coating was deposited via physical vapor deposition (PVD) in a HiPIMS process where a coating thickness of s_D = 3 µm was achieved on both WC and NbC cutting inserts. Tool performance is evaluated based on tool wear and lifetime. Coated $NbC_{1.0}$-12Ni4Mo4VC accomplished similar cutting times compared to coated WC tools without exceeding tool life criterion of VB_{max} = 0.2 mm.

2:50pm **B1-2-5 Controlled Deposition of Alpha, Beta, and FCC Tantalum Thin Films by Magnetron Sputtering**, ***Qiaoqin Yang****, S Shiri,* University of Saskatchewan, Canada **INVITED**

Due to its high chemical stability, excellent biocompatibility, and refractory nature, metallic Tantalum (Ta) has been used in many areas, including microelectronics (e.g. in capacitors), biomedical implants, surgical instruments, masks in X-ray lithography, and structural applications at high-temperatures. Metallic Ta can have three different crystal structures: body-centered cubic (alpha phase), tetragonal (beta phase), and face-centered cubic (FCC). Bulk Ta generally exists as alpha phase, which is relatively ductile and soft whereas Ta thin films obtained by vapor deposition are usually a mixture of alpha and beta phases. The beta phase, hard and brittle, is metastable and converts to the alpha phase upon heating to 750–775 °C. FCC Ta has only previously been reported as dispersed nano-grains embedded in alpha or βeta phased ultrathin films or highly deformed bulk Ta. In this presentation, we report on the controlled synthesis of Ta thin films of a few micrometers on both Co-Cr-Mo alloy and Si substrates with the three different crystal structures, including single-phased alpha Ta, single-phased beta Ta, a mixture of alpha and βeta Ta, single-phased FCC Ta, by magnetron sputtering. X-ray diffraction pattern of FCC Ta and the accurate determination of its lattice parameter are reported for the first time. Based on the results, a multilayered gradient Ta thin film, β-Ta/ β-Ta+α-Ta/ α-Ta, with high adhesion has been obtained on biomedical CoCrMo alloy sheets.

3:30pm **B1-2-7 High Power Impulse Plasma Magnetron Sputtering: Review of Critical Parameters Ensuring Successful Industrialization**, *W Gajewski, P Różański, P Lesiuk, P Ozimek,* ***AnnaWiktoria Oniszczuk****,* TRUMPF Huettinger Sp. z o.o., Poland

Since the first presentation of the High Power Impulse Magnetron Sputtering idea by *Kouznetsov and co-workers* in 1999 the basic architecture of a DC-charged capacitor bank dissipating periodically its energy into the plasma in pulses evolved to a sophisticated electronic device commercially available for industry from 2003. In order to meet rigorous requirements of industrial application, engineers have proposed different modifications of HiPIMS power delivery units to make the pulse shape and duration independent on the size of the capacitor bank and time-dependent plasma impedance.

Until now anti-wear and protective coatings prepared by HIPIMS won an established position and are used commercially. Newest market trends show the HIPIMS technology will soon became a standard production tool for oxide coatings, both conductive and non-conductive. In order to keep pace with market development HIPIMS power supplyies also requires further evolution to meet high productivity, stability and reproducibility demands of the industry. To fulfill these rigorous requirements HIPIMS power supplies must offer versatile arc management, unique control of voltage and current peak shape and the average power delivery control – sophisticated features previously unavailable in any other HIPIMS power supply units. Furthermore, those functionalities must be available both for small, laboratory size targets as well as for industrial scale where delivery of current density of 1 A/cm^2 requires a precise control of HIPIMS pulses with current of 1000 A and above.

(i) peak current regulation, *(ii)* pulse frequency, and *(iii)* pulse length.

3:50pm **B1-2-8 Investigation of the Formation of Ni-Ti Intermetallic Layers Produced by Cathodic Arc Electron-metal Ion Treatment**, ***Nagihan Sezgin****, E Kacar, K Kazmanli, M Urgen,* Istanbul Technical University, Turkey

A novel cathodic arc plasma treatment was used to obtain Ni-Ti intermetallic layers. The method was named cathodic arc electron metal ion treatment is a diffusion/coating process. During this process, AC bias was applied to the substrates and in-situ heating was achieved with electrons. AC bias potential has positive and negative cycles. At the positive cycle, electrons were directed to the substrate and the substrate was heated; at negative cycle, ions deposited on to the surface. In this study, the formation of Ni-Ti intermetallic phases were investigated for 2 different diffusion couples: Ni substrate – Ti cathode and Ti substrate – Ni cathode. Depending on the substrate-cathode diffusion couple, formed phases and sequence of the phase formation vary. To obtain desired phases should be possible by tuning the couples and process parameters. Time (5- 45 min) and temperature dependent (900 °C-1100 °C) diffusion reactions and formation sequence of phases were determined. The samples were analyzed by SEM, EDS, XRD.

4:10pm **B1-2-9 Exploring the High-temperature Stability of Nanocrystalline Cu-W Coatings**, ***Yao Du****,* Northwestern University, USA; *L Li,* Northwestern Polytechnical University, China; *J Pureza,* Universidade do Estado de Santa Catarina, Brazil; *Y Chung,* Northwestern University, USA; *K Pradeep, S Sen, J Schneider,* RWTH Aachen University, Germany

Nanocrystalline coatings are harder than their bulk or microcrystalline counterparts due to their grain size. Their hardness tends to degrade with increasing temperature due to grain growth. It has been suggested that introduction of proper alloying elements can provide thermal stabilization of the nanoscale grain structure, even at elevated temperatures. To this end, Cu-W coatings was synthesized by magnetron sputtering to investigate the grain boundary stabilization by W segregation. The thickness of the coatings is around 800 nm. The room-temperature hardness of the as-deposited coatings based on nanoindentation is 3.7 ± 0.3 GPa. Annealed at 400^{o}C, these coatings exhibit hardness decrease of 3 % after 20 minutes, probably due to stress relaxation. The hardness achieves a stable value of 3.6 GPa after two hours. The average grain size was barely changed after this extended annealing, with an average value of 36.5 nm after two hours. Atom probe tomographic analysis shows the segregation of W to the grain boundaries of Cu. These results validate the strategy for designing thermally stable nanocrystalline coatings.

4:30pm **B1-2-10 Governing the Wettability Properties of the Nanostructured Surfaces of Metallic Coatings Fabricated by Thermal Annealing***, **Feras Alzubi**, A Alkandary,* Kuwait Institute for Scientific Research, Kuwait

In this work, we report the measurement and controlling of contact angle of metallic coatings deposited by physical vapor deposition technique. We create nanoparticles on the surfaces of these coatings by thermal annealing the deposited coatings in inert environment. Thin films of Ag, Cu, Al, In, and other metals were deposited on Si substrates with 4 nm thickness. After being thermally annealed at 800 C, the ex perimental measurements of the contact angle of DIW droplets dispensed on the deposited thermally annealed metallic thin films were carried out by Optical Contact Angle system (OCA 100 Dataphysics). Our results agree with fact that the contact angle is affected by several parameters such as liquid's surface energy, roughness of the coatings' surface, type of material of the surface. Results show that the contact angle depends strongly on the type deposited material. Contact angle of all studied metals have shown a decrease towards making the surface more hydrophilic surfaces when coatings were thermally annealed. The creation of nanostructured on the surface of coatings, which was investigated by atomic force microscope, has shown to affect the hydrophobicity or hydrophilcity of the surfaces. 4 nm silver thin-film has shown a reduction from 107^o contact angle to 49.7^o making it a hydrophilic surface after thermal annealing. These findings contribute to understanding the role of metallic nanostructured on surface wettability.

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