Monday Morning, May 23, 2022

Hard Coatings and Vapor Deposition Technologies
Room Town & Country C - Session B2-1-MoM

CVD Coatings and Technologies I
Moderator: Raphael Boichot, Grenoble-INP/CNRS, France

10:20am **B2-1-MoM-2 Diamond Coatings for Cutting Tool Applications***, **Manfred Weigand (manfred.weigand@cemecon.de)**, M. Woda, W. Puetz, M. Wegh, C. Schiffers, W. Koelker, O. Lemmer,* CemeCon AG, Germany

Thin film Diamond as pure sp3 bonded crystalline carbon is able to significantly improve cutting tool performance in hard to machine applications. These coatings are typically deposited by the means of hot filament CVD processes on an industrial scale nowadays.

When applying diamond thin films to cutting tools with a cemented carbide substrate and with complex geometries, a large set of work piece materials can be machined including carbon fiber reinforced plastics (CFRP), zirconium oxides, aluminum silicon alloys, graphite or even cemented carbide. This work presents some of the latest results of various case studies revealing the benefits of CVD diamond coatings upon cutting operations on these very demanding work piece materials.

10:40am **B2-1-MoM-3 Deposition of Hard Carbon Films by High Power Pulse Magnetron Sputtering (Virtual Presentation)***, **Takayuki Ohta (tohta@meijo-u.ac.jp)**,* Meijo University, Japan*; A. Oda,* Chiba institute of Technology, Japan*; H. Kousaka,* Gifu University, Japan **INVITED**

Diamond-like carbon (DLC) films, which is an amorphous carbon including both graphite (sp2 bond) and diamond (sp3 bond) structures, has widely used for hard mask for plasma etching and hard coating of the sliding parts. The DLC film containing rich sp3 bond realizes high hardness or low friction coefficient. A bombardment of carbon ions with incident energy of 100 eV into the film is essential to increase the sp3 bond in the DLC film. However, it is difficult to produce the high energy ions in conventional direct current magnetron sputtering (dcMS). High power impulse magnetron sputtering (HiPIMS) is attractive method to obtain large ion flux or high energy ions because the peak power density of HiPIMS is about 100 times larger than that of dcMS under same average power density. A DLC film on Si substrate deposited by HiPIMS shows the hardness of 20 GPa without negative substrate bias voltage[1]. A DLC film on plastic substrate has also deposited by HiPIMS without negative substrate bias voltage. The energy distributions of argon ion (Ar^+) and carbon ion (C^+) were measured using energy-resolved mass spectrometry in HiPIMS discharge in order to investigate the relationship between hardness and ion flux. The total ion flux of HiPIMS was about 100 times larger than that of dcMS under same average power density. The distribution of Ar^+ was composed of low energy component and high energy component whereas C^+ was mainly composed of high energy component. High energy component of both Ar^+ and C^+ simultaneously increased with increasing target voltage in comparison with low energy component of Ar^+. The difference in IEDF is explained by the difference in production process of ions.

The hydrogen-free Si-DLC film was deposited by dual magnetron sputtering method using carbon and silicon targets. The friction coefficient measured with the ball on disc test decreased to be 0.074 with increasing the Si content in the DLC film. At this experimental condition, the hardness was 18.4 GPa measured by the nano-indenter. Si-C bond increased with increasing the Si content from C1s spectra of XPS analysis. The relation among sp3 fraction, hardness, and behaviors of ions will be discussed.

reference

[1] K. Iga et al., Thin Solid films, 672, 104 (2019).

11:20am **B2-1-MoM-5 Ti_3SiC_2-SiC Multilayer Thin Films Deposited by High Temperature Reactive Chemical Vapor Deposition***, **Jorge Sánchez Espinoza (jorge.sanchez@grenoble-inp.fr)**, F. Trabelsi, E. Blanquet, F. Mercier,* SIMAP, Grenoble-INP, CNRS, France

MAX phases have a unique combination of ceramic and metallic properties that make them excellent candidate materials for high temperature applications. They are particularly interesting for their excellent thermal stability up to 1500 °C, their exceptional thermal shock resistance and their damage tolerance. In addition to their good mechanical properties at elevated temperatures and high stiffness, they exhibit optical, electrical and thermal properties similar to metals.

In this work, we report on the preparation of multilayer ceramic coatings based on MAX Phases by Reactive Chemical Vapor Deposition (R-CVD), able to withstand high temperature (T>1000°C) under air while maintaining their structural and functional properties. Computational Fluid Dynamics calculations, thermodynamic and diffusion considerations were developed for the selection and the design of the multilayer experiments.

R-CVD depositions were conducted in a vertical quartz cold-wall CVD reactor in the system $TiCl_x$ - H_2 - SiH_4 - C_3H_8 - H_2 between 1100-1300 °C in order to form multi-layer coatings that consist of stacks of titanium silicon carbide (MAX phase layer Ti_3SiC_2) and silicon carbide (SiC).

The surface morphology and cross-sectional microstructure of as-grown Ti_3SiC_2–SiC layers were characterized by Scanning Electron Microscopy (SEM), and X-Ray Diffraction analysis to identify the different solid phases. High temperature oxidation tests, nano-indentation and emissivity measurements were carried out with the aim of evaluating the potential of this material for high temperatures applications.

11:40am **B2-1-MoM-6 Chemical Vapor Deposition of W(C,N): Process Parameter – Microstructure – Mechanical and Tribological Property Relationships***, **Katalin Böőr (katalin.boor@kemi.uu.se)**,* Uppsala University, Angstrom Laboratory, Sweden*; L. von Fieandt, E. Lindahl,* Sandvik Coromant, Sweden*; M. Fallqvist,* Karlstad University, Sweden*; O. Bäcke,* Chalmers University of Technology, Sweden*; R. Lindblad,* Uppsala University, Sweden*; M. Halvarsson,* Chalmers University of Technology, Sweden*; M. Boman,* Uppsala University, Sweden

There is a constant need to improve the performance of metal cutting tools. A materials research approach is the chemical vapor deposition (CVD) of coatings on the tool insert to enhance some of its properties or to introduce new functionalities. Hexagonal WC is the hard component of cemented tungsten carbide and is expected to have a good performance as a coating as well. Tungsten based coatings deposited by chemical vapor deposition are, however, so far relatively unexplored. The deposition from the gas phase can introduce new microstructures and textures, leading to enhanced mechanical properties. Moreover, the coating is expected to be under a compressive stress after post-deposition cooling if the substrate contains Co and the coating only the hexagonal WC phase. This differs from a tensile stress causing cracks in many conventional CVD coatings on cemented carbide.

The aim of this research was to deposit a new CVD coating, tungsten carbonitride (W(C,N)), which is a solid solution of hexagonal WC and WN, onto cemented carbide. Hexagonal WC and WN are line phases and are difficult to obtain by CVD due to the discrepancy in the reactivity of W-halide and C-precursors or a stable cubic WN_{1-x} phase, respectively. For the W(C,N) synthesis WF_6, CH_3CN and H_2 were used and they proved to have a balanced reactivity for W, C and N incorporation in ratios that enabled the formation of the hexagonal phase.

First, the current understanding on the growth mechanism will be presented. The results are based on a kinetic study and the influence of the process parameters on the coating microstructure and composition. Correlations between the grain size, morphology and orientation will be shown. The coating microstructure was characterized by scanning electron microscopy (SEM), the composition by elastic recoil detection analysis (ERDA) and the texture by X-ray and electron backscatter diffraction (XRD and EBSD). Transmission electron microscopy (TEM) was used to investigate a preferred prismatic texture and the type of defects introduced during the growth. Hard X-ray Photoelectron Spectroscopy (HAXPES) was used to investigate the reason behind a (C+N)-rich composition.

Further, the possibilities to tailor the microstructure and grain orientation of the coatings will be discussed. Nanoindentation measurements, diamond scratch and abrasion tests showed that the coatings were hard or superhard and their mechanical and tribological properties were dependent on the above properties.

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