Hard Coatings and Vapor Deposition Technologies
Room Golden West - Session B2-1

CVD Coatings and Technologies

Moderators: Michel Pons, University Grenoble Alpes, SIMAP, CNRS, Makoto Kambara, The University of Tokyo

4:10pm **B2-1-9 Combined Effects of Supersaturation and Stress for the Control of AlN Film Quality***, **Raphael Boichot**, D Chen,* Grenoble-INP, France*; F Mercier,* CNRS, France*; M Chubarov,* Grenoble-INP, France*; G Giusti,* Sil'Tronix, France*; M Pons,* CNRS, France **INVITED**

The aim of this talk is to show the degrees of freedom offered to CVD experimenter to tune the properties of crystal during hetero-epitaxial growth. We will develop here the example of the AlN epitaxial growth by HT-CVD with $AlCl_3$ and NH_3 as precursors in H_2 as carrier gas.

We showed in previous studies that the epitaxial growth of AlN on various substrates can be obtained by carefully aiming a particular temperature-supersaturation-growth rate process parameters window, but we also found that the AlN crystal quality, into this "epitaxial window", does not depend so strongly on the growth parameters other than growth rate. N/Al ratio surprisingly does not play a crucial role in obtaining epitaxial growth or desirable crystal quality.

Clearly to increase further the crystal quality once epitaxial growth is reached, we must cover a wider range of growth parameters to achieve a good control of crystal properties. In one hand, we will explain how mechanical characteristics of layers at room temperature can give valuable information on the first steps of growth that are critical for crystal quality and properties. In the other hand, we will show how certain growth parameters, even considered as secondary or counter intuitive, could be key to explain crystal quality and properties due to their influence on the early steps of growth.

Due to the high variability in experimental results in CVD and the need to exhaustively cover growth parameters, we will present the interest of using DOE (design of experiments) method to fasten the research in crystal growth rather that the classical one-variable-at-a-time (OVAT) studies.

Our conclusions, summarized from a huge collection of statistically robust experimental results, are that the crystal quality is the harder parameter to control compared to other properties of the grown AlN layer. In particular, the final stress state and roughness is relatively easy to tune with process parameters. We found that good crystal quality are related to high tensile stresses in the grown layers but also to certain parameters not embedded into our previous studies. We propose some explanations to the process parameters/layer properties relationship that can be generalized to other materials grown by CVD.

4:50pm **B2-1-11 Fabrication of Boron-doped Diamond Films on Cemented Tungsten Carbide***, **Kunio Saito**,* Japan Coating Center Co., Ltd.,Chiba Institute of Technology, Japan*; A Kawana,* Japan Coating Center Co., Ltd., Japan*; A Suzuki, Y Sakamoto,* Chiba Institute of Technology, Japan

To deposit diamond coating on cemented tungsten carbide is quite difficult. Generally, to reduce reactivity of cobalt which is the binder of cemented tungsten carbide that pretreatment using the acid is carried out. On the other hand, study to improve the adhesion of nanostructured diamond coating is reported, which is to formed borides by plasma enhanced chemical vapor deposition using diborane for reducing reactivity of cobalt. Diborane (B_2H_6) and trimethyl boron {B $(CH_3)_3$} is often used as a boron source. However, it is necessary to control with special equipment because of having poisonous, flammability and explosiveness of these sources.

This study is an attempt to deposit boron-doped diamond directly on cemented tungsten carbide substrates by microwave chemical vapor deposition using trimethyl borate {$B(OCH_3)_3$} as a boron source. Trimethyl borate is liquid which dissolved boric acid(H_3BO_3) in methanol(CH_3OH), and introduce to vacuum chamber as a boron source with bubbling using hydrogen(H_2) as a carrier gas. Cemented tungsten carbide substrate was scratched with diamond powders and cleaned with ultrasonic cleaner before loaded into the vacuum chamber.

Boride was formed on the substrate surface to reduce reactivity of cobalt which is the binder of cemented tungsten carbide with introducing boron source into the vacuum chamber. At this stage, confirming a combination state of the surface product by X-ray photoelectron spectroscopy(XPS), existence of boride was confirmed. Diamond was formed continuously with using reactive gases of methan and hydrogen, after forming borides on the substrate surface. In the Raman spectrum, the peak of diamond due to SP_3 was observed and the presence of diamond was confirmed. As a result of observation the surface with scanning electron microscope(SEM), it was confirmed that diamond which grew up onto substrate surface. And, boron-doped diamond with low electrical resistance approximately 10^{-2}[Ω] was obtained.

It is possible to deposit boron-doped diamond with good adhesion by introducing the bonds with boron and cobalt without pretreatment.

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