

OBTAINING OF THE CVD DIAMOND COATINGS ON THE TOOL MATERIALS

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Introduction

The crystallization from gas phase by method of the chemical vapor deposition (CVD) has many advantages. CVD synthesis makes it possible to obtain the coatings on tools with difficult geometry, but one has any negative characteristics:
1 - low adhesion to substrates;
2 - high roughness of outside diamond surface.

Poor adhesion are brought about high macrostresses occurred due to mismatch of thermal expansion coefficients (t.e.c.) by cooling from temperature of synthesis. Coating roughness is conditioned by specific mechanism growth of diamond from gas phase.

Mineralceramic and SiAlON tools are used for machining of steel and cast iron. An optimal cutting rate by mineralceramic tools is 100 - 200 m/min, by SiAlON - 50 m/min. By high temperature (1200-1400°C) in contact zone between tool and steel are formed fusible siliceous phases. They exercise negative influence on adhesive wear. The creep is developed in cutting zone at high temperature and load. This is a main factor of fracture. One of the strengthening of ceramic cutting tools is precipitation of superhard coatings with great heat-conductivity. In this paper had been analyzed the adhesion of diamond coatings on different substrates.

Materials and experimental methods

For understanding of adhesion principles of CVD coatings had been investigated the processes of the diamond deposition on various substrates: W, Si, SiO₂, B₄C, Si₃N₄, SiC, Al₂O₃, mineralceramics Al₂O₃-20%TiC-10%(ZrO₂, WC, MgO) and SiAlON. They differ in chemical affinity to carbon and t.e.c. Diamond films thickness 5-100 µm were grown of the rate 1-10 µm/h by hot-filament CVD method at total gas pressures ranging from 15 to 60 Torr, using hydrogen-methane gas mixtures consisting 0.5 - 6 vol.%CH₄, W-filament temperature of 1900 - 2200°C, substrates temperature (T_s) of 600 - 1000°C.

The crystals structure had been studied by X-ray diffraction, scanning and transmission

electron microscopy. Qualitative analysis of adhesion was making by indentation test.

Results and discussion

Kinetics of diamond nucleation depends on substrate temperature to a most extent. Mainly, its change influence on the nuclei density, size and structure of particles. The density distribution of diamond crystals in coatings is 10⁷ cm⁻² at 600°C and 10⁵ cm⁻² at 800°C. The pressure and methane concentration in hydrogen exercise on the lattice defect. The smaller are pressure and concentration CH₄, the better are diamond crystallinity. The perfect crystals grow by pressure 5 - 10 Torr and CH₄/H₂ = 0.5 - 1.0%.

By working out the CVD hot filament technique was installed that all substrates must be distributed in two parts: reacting and no reacting to carbon. From above represented substrates only W and Si have high chemical affinity to carbon therefore on the first stage are formatted carbides WC+W₂C and β-SiC correspondingly. It must be noted that β-SiC is high pressure phase which are synthesized by low pressure too. Carbide layers of thickness 1 µm have fine grained structure of size particles less than 0.5 µm. Diamond nucleation begins afterwards. Incubation period is equal nearly 1 h. This is the time of carbidizing. The degree of supersaturation decreases through carburizing of the substrate. Therefore the nucleus density on W and Si is two orders smaller than that on Al₂O₃-TiC, Si₃N₄ and B₄C by equal conditions of experiment. High density distribution of diamond particles (10⁸ cm⁻²) on these substrates led to obtain a continuous film of thickness smaller than 5 µm.

CVD synthesis had been used for deposition of strong coatings on mineralceramics and SiAlON cutting tools. Cutter was obtained by hot pressing technology. Cutting edges on tools had been machined by grinding and mechanical polish on diamond abrasive equipment. Crystallization from vapor makes it possible to obtain the coatings on the sample with difficult form. Really diamond coatings copy all morphological features of cutter. CVD coating grow on cutting edge and three sides

at the same time. But distribution and size of grains are differing: on cutting edge - 10^6 cm^{-2} and $5 \text{ }\mu\text{m}$, on the next sides - 10^8 cm^{-2} and $1 \text{ }\mu\text{m}$ correspondingly.

Potential employment of coatings depends on adhesion to substrate. It can be noted that three ways to increase of adhesion are:

1. matching of thermal expansion coefficients for minimizing of macrostresses on interphase;
2. formation of chemical combination between coatings and substrates;
3. activation of mutual diffusion.

By absence of two last factors the adhesion is defined only Van-der-Vaals forces. As a rule diamond coatings are characterized by high compressive stresses which were calculated for various substrates (Table). Stresses are originated from mismatch of t.e.c. of diamond ($\alpha = 0.8 - 1.0 \times 10^{-6}/^\circ\text{K}$) and substrates, high temperature of synthesis and maximum Young's modulus of diamond.

Table

Calculated thermal macrostresses (σ) in diamond coatings on substrates by cooling from $T_s=850^\circ\text{C}$

Substrate	$\alpha(10^{-6}/^\circ\text{K})$	$\sigma(\text{GPa})$
Si	2.6	1.7
β -SiC	4.7	3.7
W	4.4	3.3
WC	3.8	2.7
SiAlON	2.7	1.7
Al_2O_3	6.3	5.2
Al_2O_3 -20%TiC	7.7	6.6

As shown X-ray diffraction the diamond coating and mineralceramics are not chemical interaction and mutual diffusion. Adhesion was tested by Vickers indentation method. Under a load of 3N the cracks are appeared on interphase boundaries therefore diamond coatings are free standed from mineralceramics. The reason for poor adhesion is large thermal stresses ($>6 \text{ GPa}$).

SiAlON are not reacted to diamond. Level of stresses is nearly equal the same on substrates W and Si but adhesion bond is maximum. Under indenter are not observed the delamination. It can be supposed that strengthening occur due to interfacial diffusive nanolayers formatted by carbon diffusion into bonding phase in SiAlON.

Conclusion

There are studied the technological and structural aspects of hot filament CVD diamond growth on mineralceramics $\text{Al}_2\text{O}_3 - 20\% \text{TiC} - 10\%$ (ZrO_2 , WC, MgO) and SiAlON. Depending on the degree of supercooling were produced coatings with polyhedral or globular structure. Distribution of crystal grains is differing on cutting edge and three sides of tools. The roughness of diamond coatings on SiAlON is smaller than that on the mineralceramics, but adhesion is bigger. Calculated compressive stresses of diamond on various substrates are 1-7 GPa. They can not be relaxed by means mechanical coupling. High adhesion is obtained thanks to chemical interaction of diamond coatings and substrates.