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EFFECT OF HEAT TREATMENT ON PHYSICAL AND MECHANICAL PROPERTIES OF CVD DIAMOND

ВПЛИВ ТЕРМІЧНОЇ ОБРОБКИ НА ФІЗИКО-МЕХАНІЧНІ ВЛАСТИВОСТІ CVD АЛМАЗУ

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Abstract. *The effect of heat treatment on the physical and mechanical properties of CVD diamonds at both normal and high pressure is investigated. The results indicate that these properties are significantly influenced by the structural characteristics of CVD diamonds. Specifically, the hybrid material reinforced with black CVD diamond exhibits the highest thermal stability, with a mass loss of only 0.9%. After heat treatment, CVD diamond samples lose between 17% and 48% of their initial weight, depending on their structure. The strength of the samples did not change significantly before or after HPHT treatment, but decreased sharply (by approximately 18 times) after heat treatment in an argon environment at normal pressure.*

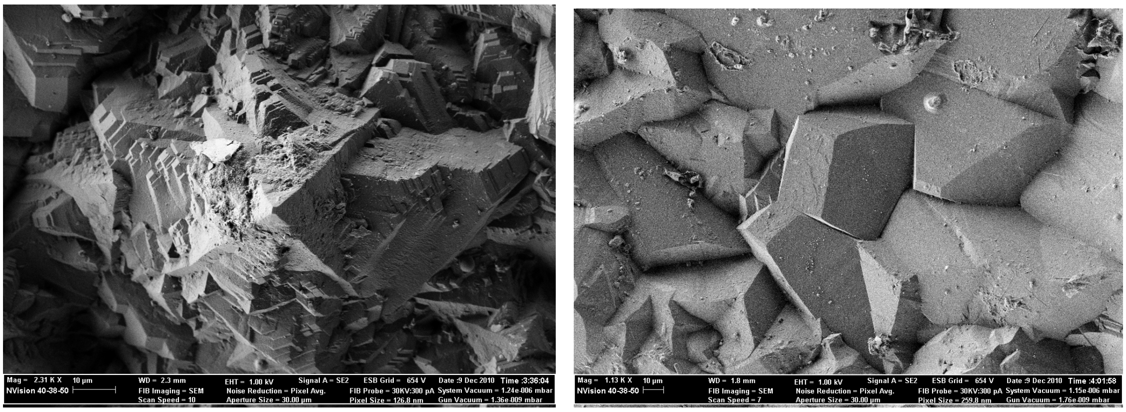
Key words: *diamond, Chemical Vapor Deposition, heat treatment, strength*

Introduction.

Diamonds produced by chemical vapour deposition (CVD) began to spread rapidly in the early 1980s when it became possible to produce them on an industrial scale. CVD diamonds are similar in properties to single diamond crystals. With sufficiently pure reaction gases, the nitrogen content (the main impurity in natural and synthetic crystals) in CVD diamonds can easily be reduced to 1 ppm. Growth rates for different deposition processes can vary considerably and it is usually the case that higher growth rates can only be achieved at the expense of a corresponding loss in CVD diamond quality [1]. As an example, Figure 1 shows microelectron images of fragments of the growth surface of 4×1 mm and 0.4 mm thick CVD diamond wafers grown at different growth rates, taken with a JEOL JSM-6480LV low-vacuum scanning electron microscope (SEM).

Today, polycrystalline single crystal CVD diamond films and wafers with diameters exceeding 100mm and thicknesses ranging from microns to 1-3mm are grown using the CVD process at temperatures of 700-1000°C and operating pressures of 30-100 Torr.

Over the last 10-15 years, the development of the CVD process has made this unique material available and in demand in many areas where the extreme properties of diamond are required. One of the most important areas of modern CVD diamond research is the development of technologies for the production of polycrystalline diamond for tooling, in particular for the production of CVD reinforced components of hybrid materials.



a b

Figure 1 - Images of fragments of the growth surface of CVD diamonds grown at different growth rates

As you know, a hybrid is an object that combines the properties of other (two or more) objects. In engineering, the adjective "hybrid" is used to emphasise systems that contain different elements or processes [2]. The peculiarity is that the respective elements are complete solutions and as a result of their combination new desired properties are created.

At present, a new direction of superhard material production is being actively developed - hybrid diamond based polycrystalline superhard composites [3]. In particular, a hybrid diamond-based superhard material (trade name "Hybridite" [4]) has been created, which is a plate of mono- or polycrystalline CVD diamond encased in a polycrystalline envelope of a heat-resistant diamond composite material (DCTM). This combines the unique physical, mechanical and thermal properties of CVD diamond with the high hardness, strength and heat resistance of a polycrystalline shell of static synthesis diamond. The effectiveness of CVD diamonds in tools is primarily determined by their physical and mechanical properties, particularly strength and thermal stability, so knowledge of the strength properties of CVD diamonds is of great scientific and applied importance.

The aim of this work is to investigate the effect of heat treatment on the physical and mechanical properties of CVD diamond.

Main text.

In order to investigate the effect of heat treatment on the physical and mechanical properties of CVD diamond, samples were taken both in the form of bars and in the form of a 630/500 grit powder. The samples differed both in colour (light and black bars, light and grey powder particles) and in structural perfection.

The colour of the CVD diamond samples was determined by their degree of purity and structural perfection, which depend on the synthesis technology. The so-called "black diamond" is a polycrystalline CVD diamond with a high content of structural defects such as microtwins, dislocations and nanometre amorphous domains [5]. These defects cause strong optical absorption in the so-called "black" diamond, while graphite inclusions are absent. However, during vacuum annealing of polycrystalline diamond, even if initially transparent, to temperatures above 1200-1300°C, thin layers of crystallised graphite, several interplanar distances thick, can form at the grain boundaries [6], which also leads to blackening of the material.

To determine the strength of CVD diamond rods under uniaxial static compression, samples measuring 1.0×1.0×0.4 mm were taken. The germinal side of the bar was smooth (mirror) and the opposite (growth) side was rough (Figure 2).

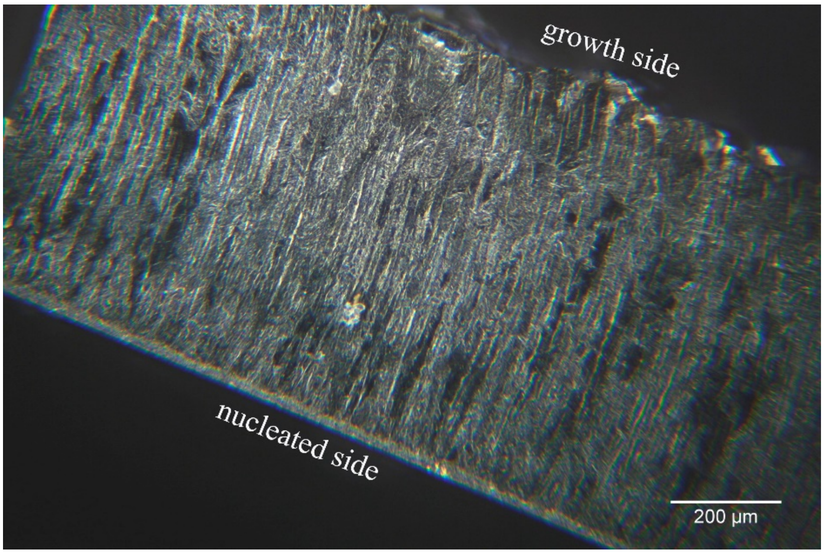


Figure 2 – A CVD diamond bar measuring 1.0×1.0×0.4 mm.

A 0.02 mm thick copper foil was placed on the growth side to maintain plane-parallelism.

Heat treatment of CVD diamond rods and powders was performed in a muffle furnace in an argon stream at a temperature of T = 1150 °C for 600 s. This heat treatment regime corresponded to the conditions of tool manufacture. This heat treatment regime corresponded to tool manufacturing conditions.

CVD diamond rods processed under high pressure and high temperature (HPHT) conditions were also studied – at 7.0 GPa and 1350 °C in a graphite medium (grade C-3) for 60 s.

A WPM-1000 tensile tester was used to determine strength. The scale used in the test was 0–500 kg. The destruction was carried out between two supports made of VK2M hard alloy and the speed of the loading programme was 40 kg/s.

The obtained uniaxial compressive strength of CVD diamond rods is shown in Table 1.

Table 1 - Uniaxial static compression strength of CVD diamond plates with dimensions of 1.0×1.0×0.4 mm

CVD diamond sample	Crushing load, kN	The type of destruction
Initial	1,7±0,2	When the critical CVD load is reached, the diamond breaks into small fragments
After HPHT treatment (p=7.0 GPa; T=1350 °C) in a graphite medium	1,7±0,2	Similarly
Після термічної обробки в струмі аргону (T=1150 °C)	0,09±0,02	When the critical CVD load is reached, the diamond breaks into larger fragments

From the above data it can be seen that the strength of CVD diamond samples, both initially and after NRNT treatment, has not changed and after heat treatment in an argon environment it has decreased dramatically (~18 times).

The strength of CVD diamond powders with a grit size of 630/500 was also studied (Fig. 3).

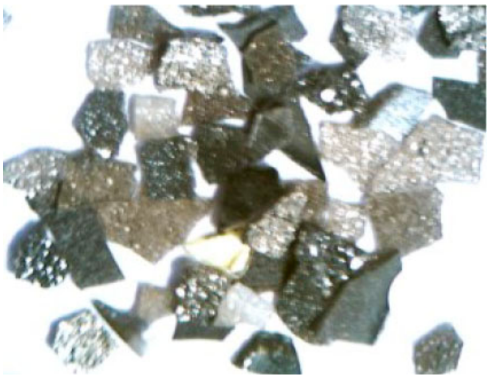


Figure 3 - CVD diamond powder with a grit size of 630/500

As can be seen in Figure 3, the powders consist of light and grey grains. In order to study the effect of the colour of the CVD diamond grains on their strength, they were conditionally divided into "light" and "grey" grains.

The strength values obtained for uniaxial static compression of CVD diamond grains are shown in Table 2. Before measuring the strength, the grains were conditionally divided into "light" and "grey" grains.

Table 2 - Uniaxial static compression strength of CVD diamond with 630/500 grit (grain thickness 0.23 mm)

Crushing load, kN			
"Light" grains		"Grey grains	
Initial	After heat treatment	Initial	After heat treatment
1,72	1,80	1,45	1,80
2,0	2,87	2,12	1,15
1,53	1,87	0,53	0,45
2,15	2,01	0,75	0,85
2,65	1,05	0,96	1,05
1,90	1,84	1,25	1,14
1,85	2,96	2,04	1,72
1,64	1,90	0,67	0,58
2,02	0,95	0,83	0,75
2,38	2,12	0,91	0,96
Average value of the crushing load, kN			
2,0±0,2	1,9±0,5	1,2±0,4	1,0±0,3

As can be seen from the data in Table 2, the average grain strength after heat treatment remained practically unchanged.

The change in the mass of different CVD diamond samples after heat treatment in argon current at a temperature of T = 1150 °C for 600 s is shown in Table 3.

Table 3 - Weight change of CVD diamond samples after heat treatment in argon current (T = 1150 °C; duration - 600 s)

Sample	Mass of the material, g×10^{-2}		Change in mass, %
	initial	after thermal treatment	
Light CVD diamond, size 5×0.8×0.8 mm	6,20 (1 sample)	–	not determined
Balck CVD diamond, size 4×1×0,3 мм	2,74 (3 samples)	2,68	–2,2
Yellow CVD diamond, size 1×1×0,4 mm	0,63	0,07	–88,9
Light yellow CVD diamond (pieces)	1,66	1,09	–34,3
CVD diamond powder, grit 630/500	2,03	1,72	–15,3
Hybrid material (black CVD diamond + polycrystalline diamond shell)	12,58	12,46	–0,9

The most heat-resistant CVD diamond bars are black (weight loss ~2.2%).

When used to reinforce the insert, the weight loss of a CVD diamond after heat treatment is reduced to 0.9%.

Conclusion.

Thus, the results of the study of the effect of heat treatment on the physical and mechanical properties of CVD diamonds have shown that the latter depend significantly on the structural characteristics of CVD diamonds. In particular, it has been shown that the hybrid material reinforced with black CVD diamond has the highest thermal stability, with a mass loss of 0.9% for these samples. CVD diamond samples lose between 17 and 48% of their initial weight after heat treatment, depending on their structure.

The strength of CVD diamond samples, both initially and after NRNT treatment, remained practically unchanged, and after heat treatment in an argon environment at normal pressure it decreased sharply (~18 times).

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Анотація. *В роботі розглядається вплив термічної обробки як при нормальному, так і при високому тиску на фізико-механічні властивості CVD алмазу Показано, що вони суттєво залежать від структурних особливостей CVD алмазу. Зокрема встановлено, що найбільш високу термостабільність має гібридний матеріал, армований чорним CVD-алмазом (black diamond). Втрата маси для цих зразків становила 0,9%. Зразки CVD-алмазу після термічної обробки в залежності від їх структури втрачають від початкової маси від 17 до 48%. Міцність зразків CVD алмазу як вихідна, так і після HPHT-обробки, практично не змінювалася, а після термообробки в середовищі аргону при нормальному тиску – різко зменшилася ~18 разів).*

Ключові слова: *алмаз, хімічне осадження з газової фази, термообробка, міцність.*

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