

**KINETICS OF THE DENSIFICATION OF POLYCRYSTALS
SINTERED BY THE HPHT METHOD FROM DIAMOND
POWDER SYNTHESIZED IN THE SYSTEM
Mg–Zn–B–C COATED BY TITANIUM**

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The dependence of the density of diamond polycrystals sintered at different temperatures by HPHT method from diamond powders synthesized in the Mg–Zn–B–C system on the duration of sintering was investigated. The purpose of this work is to use titanium as a protective coating on the surface of diamond powder particles to create an insulating layer during sintering between the oxygen contained in the pores during sintering and the surface of the diamond powder, to reduce the degree of transformation of diamond into graphite. The density and porosity of the sintered polycrystals were measured by hydrostatic weighing. The optimal temperature and duration of sintering for diamond particles synthesized in the Mg–Zn–B–C system with a titanium coating were determined, they are, respectively, 1500°C and 20–30 seconds.

Keywords: diamond powder, composite, HPHT method, graphitization

The sintering of diamond polycrystals and composites is realized by the High Pressure High Temperature – HPHT method, since without high pressure, diamond graphitizes. However, even in such conditions, due to the air present in the pores between the grains, oxygen is also present, which interacts with the diamond during sintering, and thus causes undesirable degradation of the diamond.

The idea of this work is to use diamond powders, the surface of which is protected by a titanium coating, to create diamond polycrystals. Sensitive characteristics to the sintering process are the density and porosity of the obtained materials. In this work, the density and porosity of polycrystals sintered by the HPHT method from titanium-coated diamond powders synthesized in the Mg–Zn–B–C system were studied. The pressure in the high-pressure apparatus during sintering of the composites was 7.7 GPa, the mass fraction of titanium in the finished mixture of diamond powder was 1.5%.

The density was determined by the method of hydrostatic weighing. Open porosity was calculated as the ratio of the volume of water remaining in the pores of the sample to the volume of the sample. Total porosity – one minus the ratio of the density of the sample to the value of the diamond single crystal – 3.53 g/cm³. When calculating porosity, the presence of titanium can be

neglected, since the effect of titanium does not exceed 0.5% of the deviation from the calculation results. The measurement results are presented in Table 1.

It is worth noting that at a temperature of 1500°C, samples obtained from diamond powders of the growth system Mg–Zn–B–C, coated with titanium, have the maximum value of density.

Table 1. Porosity and density of samples at different sintering temperatures.

№	Temperature T, °C	Sintering time t, s	ρ , g/cm ³	Total porosity, %	Open porosity, %
1	1300	20	3.36	4.92	3.17
2	1500	20	3.45	2.18	0.72
3	1650	20	3.42	3.08	0.28
4	1850	17	3.29	6.82	1.10

For comparison, at the optimal sintering temperature (1650°C) of diamond powders not covered with titanium, the density is 3.37 g/cm³ [1]. The density values of samples 1 and 4 are low. In the first sample, the sintering temperature is not sufficient for plasticization and densification, as a result of which the value of open porosity is high. Sample №4 has lower density values due to graphitization, at the same time the pores are closed with graphite and, accordingly, the open porosity has a smaller value.

Comparing the porosity of samples № 2 and № 3, we note that the share of open porosity in sample № 3 is lower than in sample № 2. It can be assumed that the reason is graphitization, which is higher in sample №3 compared to №2.

Subsequently, a temperature of 1500°C degrees was chosen as the optimal sintering temperature, which can be easily seen, based on the results of Table 1, and a study of the kinetics of compaction of diamond powder at this temperature was conducted. The results of the research are shown in Table 2.

Table 2. Density and porosity of samples at different sintering times at a constant temperature.

№	Temperature T, °C	Sintering time t, s	ρ , g/cm ³	$\frac{\rho_s}{\rho_{mnc}}$	Porosity, %
1	1550	5	3.28	0.929	7.1
2	1450	10	3.44	0.974	2.6
3	1500	20	3.45	0.977	2.3
4	1450	30	3.44	0.997	0.3
5	1500	45	3.41	0.966	3.4

It is easy to see from Table 2 that the optimal sintering time for diamond powder synthesized in the Mg–Zn–B–C system with a titanium coating is 20-30 seconds. During sintering for a shorter duration, the compaction process of the diamond composite does not reach a maximum, but the graphitization of

diamond particles is minimal. On the contrary, when sintering with a time longer than 30 seconds, increasing amount of graphitization of diamond powder is added to the completed compaction process, this reduces the overall density of the composite, which can be seen in the figure 1.

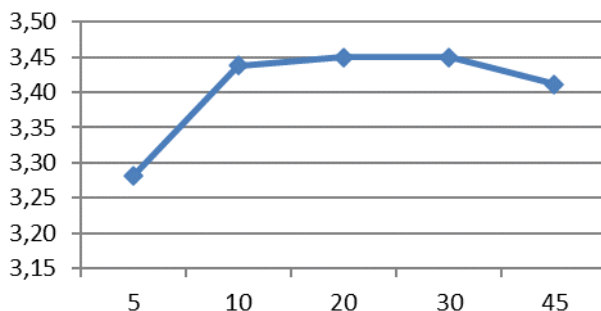


Fig. 1. Dependence the density of diamond polycrystals on the sintering time at temperature 1500°C.

According to the Fig. 1. for a duration of sintering longer than 10 s, the density of polycrystals sintered from diamond particles synthesized in the Mg-Zn-B-C system and coated with titanium reaches a "plateau", this continues up to 30 seconds, and then it decreases, which, presumably, due to graphitization of diamond powder.

In view of the above, it can be stated that titanium applied to the diamond surface significantly affects the sintering process, intensifying it and slightly reducing the amount of graphitization. For a deeper understanding of the processes that occur during sintering, it is necessary to carry out further studies of the obtained samples by the methods of microstructural and X-ray phase analysis.

1. Chernienko O.I., Bochechka O.O., Lutsak E.M., Belyaev A.S., Klepko O.Yu. and Lysovenko S.O. Thermal conductivity and electrical resistance of diamond polycrystals and diamond-copper, diamond-copper-titanium composites // Rock-destroying and metal-working tools - technique and technology of its manufacture and application. – 2018. – № 21. – P. 258–267. (in Ukrainian)