THE COMPRESSION NATURE OF THE „WURTZITE BORON NITRIDE-DIAMOND" SINTERED UNDER HIGH PRESSURE

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Abstract: *The results of experimental studies of the compression process during the sintering under high pressure conditions of wurtzite boron nitride and diamond powders mixture of submicron sizes obtained by different technologies are given.*
It is determined that the compaction level at obtaining compositions depends on the dispersion and nature of the diamond component.
Interaction of wurtzite boron nitride with diamond proceeds more intensively with diamonds of dynamic synthesis, but it is accompanied by their partial graphitization.
KEY WORDS: *WURTZITE BORON NITRIDE, DIAMOND, DISPERSION, SINTERING, DENSITY.*

Introduction.

A considerable amount of work is devoted to obtaining composite polycrystalline materials on the basis of superhard phases of boron and carbon nitride. In experiments [1-4] mixtures of boron and carbon nitride were used, in which one of the components was used in the state of thermodynamic stability under high pressure conditions.

However, the production of composites from such mixtures requires very high barometric parameters for sintering and direct phase transformations in dense modifications of components with a layered crystalline lattice, therefore, in [1], when composite from a mixture of hexagonal boron nitride and diamond was obtained, to decrease barothermal parameters, catalytic impurities were used.

In addition, a number of papers [3-5] reported the obtaining of "CBN-diamond" composite materials when graphite-like boron carbonitride powders [3, 4], a mixture of diamond and graphite-like boron nitride [5] or diamond and sphalerite boron nitride [6, 7] were used.

In view of the fact that any data on the nature of the compression of said powder systems under the influence of high pressures and temperatures, there are no studies in this direction that are of scientific interest.

The purpose of the research is to study the peculiarities of formation in conditions of high static pressures and temperatures of polycrystalline composite materials in the system "wurtzite boron nitride - diamond".

Methods of experimental research. As the main component of a powder system mixture for the composite material production, the wurtzite boron nitride powder, which meets the requirements of TUU 75-12006.7-98, was used.

The main fraction of particles had a size in the developed plane of 0.1 ... 5 microns. According to electron microscopic studies, the powders were fragmented at two levels: the size of the first-level fragments was 0.5 ... 1.0 μm, and the second level was 50 ... 100 nm or less.

Diamond powders of different dispersion and origin were used as the second component of the mixture to produce the composite material - a catalytic synthesis of submicron sizes of 0.1/0 μm and dynamic nanosized range 0.005 ... 0.012 μm.

To study the compression kinetics and contact interaction between the system components, wurtzite boron nitride and diamond at sintering under high pressure, a mixture of their powders was prepared in a quantitative weight ratio of 90:10, from which pressed samples of a cylindrical shape.

The obtained samples in the high-pressure apparatus of the "toroid" type were subjected to high pressure p = 7.7 GPa and temperatures T = 1500 ... 1800 °C at an interval of 100 °C for 5, 15, 30, 60 and 120 seconds.

The temperature control was carried out in accordance with the schedule of thermocouples previously obtained in the coordinates "power of heating – temperature".

At the beginning of the sintering process, the instant of heating was taken - non-isothermal conditions prior to the establishment of a quasi-stationary thermal regime.

The density of the sintered samples was determined by hydrostatic weighting method using the WLR-200 analytical scales with a maximum weighing range of 200 g and an accuracy of ± 0.15 mg.

Discussion of research results.

An analysis of the density measurements of sintered samples of the "wurtzite boron nitride-diamond" system composite material suggests the following.

According to studies of the compression nature of the initial mixture during the sintering of composite materials, it is observed its dependence on the origin and dispersion of the diamond component.

Despite the fact that the samples density from mixtures of boron nitride and diamonds of different origin in the initial state before the temperature action had approximately the same value, the compression nature of compositions is significantly different.

The samples density containing diamonds of static synthesis of 1/0 μm at the initial stage of sintering under nonisothermal conditions already after 3 ... 5 s significantly exceeds the characteristics of the composition with diamonds of explosive synthesis (ρ_{st} = 2.68 ... 2.84 g/cm^3 against ρ_{dyn} = 2 , 16 ... 2.31 g/cm^3).

In the whole range of temperatures at all stages of sintering over τ = 60s, a gradual increase in sample density to a maximum value of ρ = 3.412 g/cm^3 is observed.

Figure 1 shows the compression kinetics of the powder composition "wurtzite boron nitride - diamond" at sinterng under high pressure conditions.

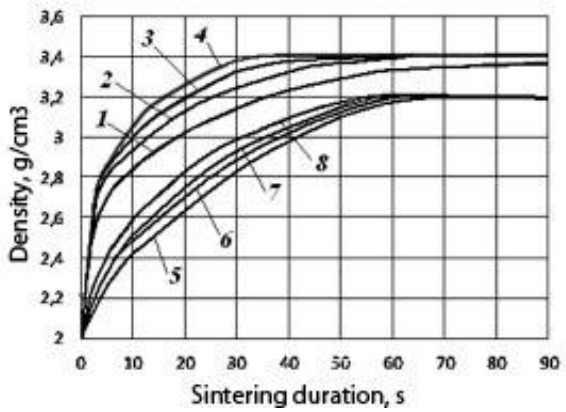

Figure 1. *Compression kinetics of a powder composition, at sintering under high pressure conditions (p = 8 GPa): BN_w + static synthesis diamonds at T = 1500ºC (1); T = 1600ºC (2); T = 1700ºC (3); T = 1800ºC (4) and BN_w + dynamic synthesis diamonds at T = 1500ºC (5); T = 1600ºC (6); T = 1700ºC (7); T=1800ºC (8).*

The maximum density of the sintered composition "BN_w - Explosive Synthesis Diamond" was ρ = 3.256 g/cm^3, and the nature of its change, depending on the sintering duration, is similar to

composition with static synthesis diamonds, but the sample density at all stages of sintering is significantly lower.

This is due to the fact that already at T = 1000 ºC there is a graphitization of dynamic synthesis diamonds[8] and, according to the data of X-ray analysis [9], at T = 1600 ºC the main part (~ 70%) is completely graphitized, while the other (~ 30%) forms a solid solution with boron nitride during the phase transformation of the wurtzite modification of BN into sphalerite [10].

It is the porosity that occured as a result of the phase transformation of diamonds into graphite, which determines the characteristics of the sintered composition samples with the indicated density indices, and its volume corresponds to the mass fraction of diamonds that have been graphitized.

It was shown in [11] that at sintering diamond powders with particle sizes smaller than 0.3 μm already at a temperature T = 1600 ºC, as a result of their graphitization, the amount of counterweight in the pores of the samples is comparable to the value of the external pressure (p = 8 GPa).

Accordingly, the results of our X-ray diffraction analysis of the matrix component of the composite based on wurtzite boron nitride are obtained.

The level of phase transformation $BN_w \rightarrow BN_{sf}$ in the composition of the explosive synthesis diamond under the same barothermic conditions, lower than the compositions based on static synthesis diamonds, due to pressure drop in the pores of the graphite, which is applied to the total pressure in the volume of samples and contributes to the intensification of the phase transformation of $BN_w \rightarrow BN_g$ in the case of discrepancy in the volume of samples of the thermodynamic stability conditions of high pressure nitride boron phases.

It is significant that for both systems of composition the maximum density of sintered samples is achieved at the temperature T = 1700 ºC.

Such temperature of sintering superhard materials based on wurtzite nitride boron type composite-10 have the highest physical and mechanical characteristics, and in the given composition the main component is wurtzite boron nitride, which determines the nature of its formation.

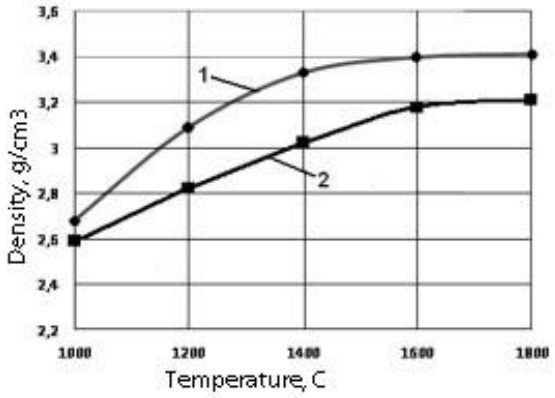

Figure 2. *Compression characteristics of powder composition "wurtzite boron nitride - diamond" at sintering under high pressure conditions (p = 8 GPa):*

BN_w + static synthesis diamond 0,1/0 μm (1);
BN_w+ dynamic synthesis diamond (2).

The increase of the sintering temperature of the composition to T = 1800 °C and the duration of sintering up to 120s for both systems leads to a decrease in the material density, due to the peculiarities of the structure evolution of the polycrystalline material on the basis of BN_w during the sintering process, when the polycrystal dissolution occurs due to the development of plastic deformation by creep, which is associated with the formation of microstructure sections of the material on the monophase grains BN_{sf} basis with well-formed boundaries between them [12].

The compression process of the powder body is fully controlled by the development of plastic deformation, which mechanisms change in accordance with the evolution of the structural and phase state in the system of particles.

The fast packing compression state (initial stage) of sample is determined by the cooperative plasticity, which determines by the anisotropy of the lattice of wurtzite boron nitride and the insignificant energy of the basic packaging defects and the disordering of the initial BN_w due to the accumulation of basic defects in the packaging, which is accompanied by an increase in plasticity [13].

Cooperative deformation affects the formation nature of samples density, due to the next stage of structure formation - dissolution of the material.

Conclusion.

The results of the conducted studies indicate that the dispersion and nature of diamonds in the composition of the wurtzite boron nitride determine the characteristics of the polycrystalline material compression during the sintering under high pressure conditions. In the presence of static synthesis diamonds of submicron sizes, the compression process of the powder system during sintering corresponds to the general laws for wurtzite boron nitride in its pure form.

For composition with dynamic synthesis diamonds, the level of components compression of the composition is limited by partial graphitization, which is approximately 30% of the initial amount of the diamond component.

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