

Consolidation of superhard particles at high pressures and high temperatures

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Consolidation of diamond or diamond-like boron nitride particles is carried out in the P-T region of their thermodynamic stability. Typically, a pressure range from 5 to 8 GPa is used. But even under these conditions, at temperatures above 1500 °C, the binding of individual particles into a solid compact is impossible due to the reverse phase transformation of the surface layers into graphite-like phases. Therefore, consolidation is carried out with different activating additives. Metals of iron group [1,2] or components (silicon, titanium), which form refractory carbides are used in the synthesis of diamond composites. Binderless nanopolycrystalline diamond compacts are obtained by the direct transition of graphite sample at pressures of about 15 GPa and temperatures above 2000 °C [3]. Its hardness is greater than 100 GPa, but the samples sizes are limited with several millimeters.

Diamond-like boron nitride (cBN) has lower hardness than diamond, but the former one has higher thermal stability. Strong compacts can be obtained by sintering of mixtures of cBN microcrystals with nanopolycrystalline particles of wurtzite modification (wBN) [4] without activating additives. It requires the pressure of about 8 GPa, so it seems that we need to find the most perspective way how to bind diamond particles with boron nitride ones. It is necessary to create P-T conditions to avoid surface graphitization as well as to form well-bonded grains of diamond-diamond and cBN-diamond crystallites during composites synthesis. This paper presents a new approach to get consolidation of micro and nano superhard particles.

Synthetic diamond powder with particle size less than 100 nm was used as a starting material. This powder was mixed with melamine (C₃N₆H₆) and M-carborane (B₁₀H₁₆C₄O₂). Experiments were performed in toroid type chamber in the pressure range within 7-8 GPa and temperature range within 1600-1700 °C. The pyrolysis of melamine and carborane takes place under heating. Reaction between boron and C-H, N-H fluids leads to formation of carbon doped diamond-like boron nitride and boron doped diamond crystals.

Conducted earlier structure refinement using the Rietveld method showed that up to 10% of carbon atoms can be in crystals with a basic lattice of boron nitride, and the maximum increase in the unit cell size is about 1%. Carbon occupies the positions of both boron and nitrogen [5]. In boron-doped diamonds, the unit cell parameter has two discrete values: about 3.570 Å for small boron concentrations (~1-1.5%) and about 3.578 Å

for higher ones (~2-3%). Meanwhile the number of vacancies in the diamond lattice exceeds the concentration of boron atoms in 2-3 times [6].

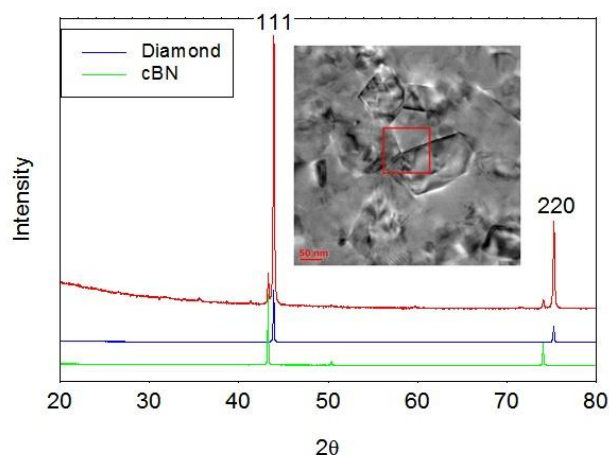


Figure 1. X-ray diffraction pattern and TEM image of diamond-cBN composite.

X-ray phase analysis of the samples synthesized from mixtures of nanodiamonds with melamine and M-carborane confirmed the absence of graphite-like formations. TEM analysis of microfoils showed a dense stacking of superhard particles in the composite (Fig. 1). Well-bonded grains and perfect borders were observed between cBN-diamond and diamond-diamond crystals.

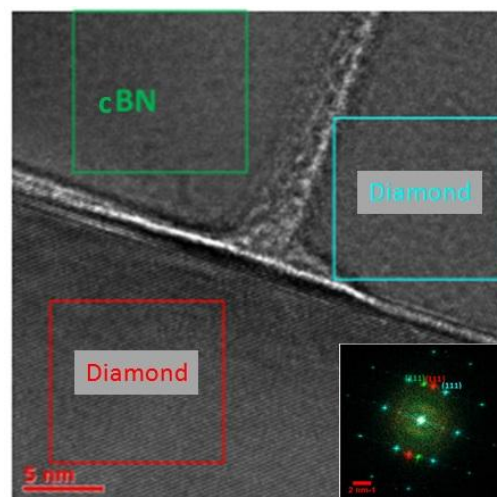


Figure 2. Fine structure of the interface between crystallites of diamond and cBN.

The process of consolidation of nanodiamonds took place by means of fluid phases, that facilitated the mutual movement of particles and provided an active mass transfer on the surface. A very high concentration of point defects (substitution atoms and vacancies) in the volume of diamond and cBN crystals, which were formed directly during the thermobaric treatment, contributed to the activation of diffusion processes and the formation of perfect interfaces as well (Fig. 2).

The considered approach can be used for the synthesis of superhard composites without binder. Necessary for its implementation thermobaric parameters are acceptable for large-scale reproduction.

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