THE POSSIBILITY OF SYNTHESIS OF BORON CARBIDE PHASE

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A boron carbide compound was discovered in 1858. The 'stoichiometric formula' B_4C was only assigned in 1934 [1]. Boron carbide is an interesting material in many of potential applications, such as an abrasive and superhard material [2], a thermoelectric device material [3], control rods for nuclear power generation and as a neutron radiation absorbent [4,5]. Boron carbide is one of the lightest and hardest known materials and as such is particularly suited to application as an armor ceramic. Boron carbide is nominally assigned the formula B_4C, although it has a wide range from B_4C to $B_{10.5}C$ and corresponding to a composition range of about 20–9 at.%C, respectively [6]. There are a lot of methods of obtaining a boron carbide powder and film. They might be both industrial methods and laboratory methods of synthesis [1]. And this article presents a potential method, which can help to obtain boron carbide nanostructures. This method is based on coaxial magnetoplasma accelerator (CMPA) developed at a scientific laboratory of National Research Tomsk Polytechnic University [7].

The using of CMPA shows the possibility of dynamic synthesis for creation of super dispersed crystalline phase in hyper speed jet boron carbide electric–discharged plasma. The plasma steams into a hermetically sealed volume of a chamber–reactor with an argon atmosphere at a pressure of 5 atmospheres. The source of the plasma is high-current (about 10^5A) impulse (0.5ms) CMPA with a graphitic accelerator channel (AC) and a central electrode.

Power source is obtained from a capacitor, which has volume of C=6 mF. Charge voltage is about 3 kV. Dispersed carbon (soot) and boron precursors are placed in current-increasing Z-pinch plasma. Furthermore, carbon electro-erosion from accelerator channel occurs. In plasma discharge a target material is being intensively sputtered. As the result, quenching provides the nanodispersibility of synthesized phases. It is emphasized about extreme conditions for synthesis of the unique boron carbide nanostructures in the experimental process.

The phases of the synthesized powder were examined by X-ray diffraction (XRD; diffractometer Shimadzu 7000s) using CuKα radiation. Particle size and morphology analysis were performed using transmission electron microscopy (TEM; Philips CM30). The results of these analyzes show the desired boron carbide phase.

Structural characterization using transmission electron microscopy (TEM) is depicted in Fig. 1. The most peaks can be indexed to the rhombohedral boron carbide. There are three types of the nanoparticles in the powder: with the dimensions less than 1 μm, with the dimensions between 100 nm and 200nm and with the dimensions less than 100 nm.

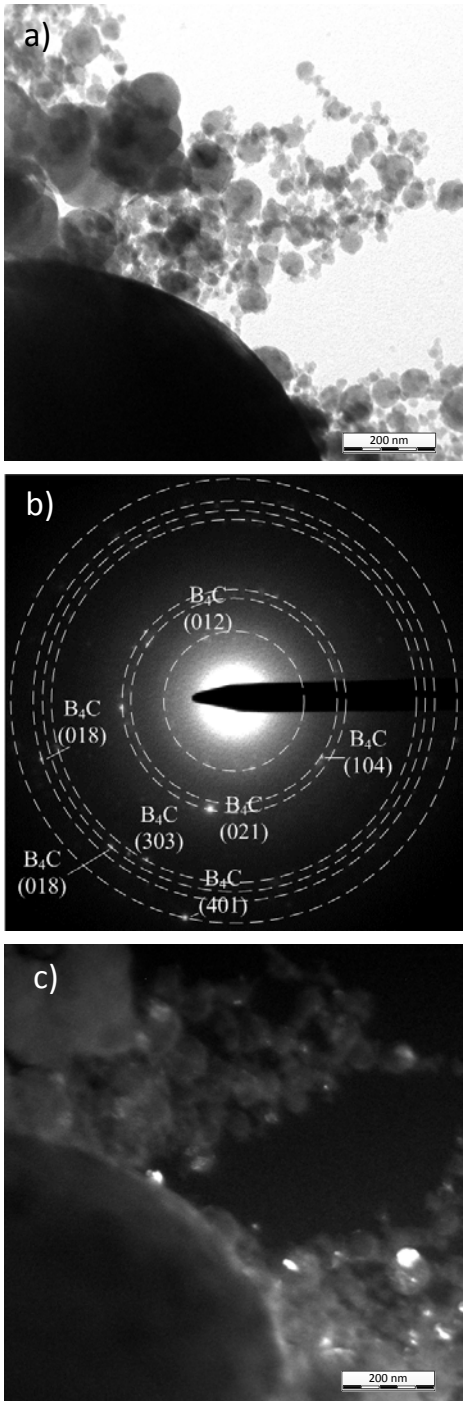

Fig.1. The data of the electron microscopy of the product synthesized: a) bright field TEM micrograph of a dispersed concentration of the particles b) selected area electron diffraction (SAED) c) dark field TEM micrograph of the dispersed concentration of the particles

Thus, the most nanoparticles are round and relatively large objects (from 20 nm to 200 nm) and supposedly they are the boron carbide nanoparticles. Also there has been the nanoparticles less than 100 nm with indiscernible morphology.

The SAED pattern emphasizes the boron carbide content by the diffraction spots 021, 104 and 220. The diffraction spot of boron carbide 104 is identified in the Fig. 1b) and the dark field TEM micrograph is made. The dark field micrograph of a typical indentation reveals shear bands oriented along the 104 directions.

The result of the XRD analyses is presented in the Figure 2. And it illustrates that there are several super dispersed crystalline phases in the obtained composition, such as boron carbide, silicon carbide and carbon.

The analysis of dynamic synthesis product is made by diffractometer Shimadzu XRD 6000, a special program PowederCell 2.4 and data base PDF4+. All possible structures of crystalline phases which can appear in the considered system are being used when the phase analysis is carried out. When there is a superposition of the following phases boron carbide (R -3m space group), silicon carbide (F-43m space group), graphite (P6-3mc {186} space group) the least divergence is received.

Table 1 presents the results of the XRD analysis and a percentage of boron carbide is about 97 (B_4C), 0.5% - SiC and 2.5% - carbon.

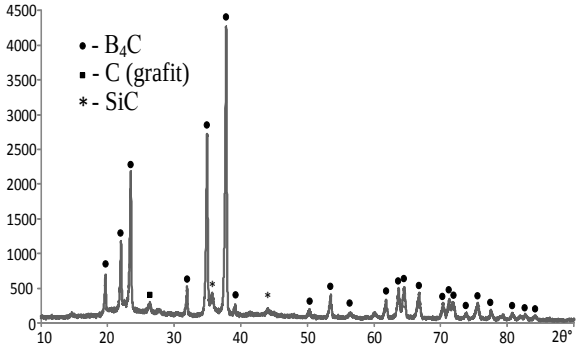

Fig. 2. XRD patterns of the obtained product

All crystalline phases in the observed product have nanostructure and it is provided by average coherent-scattering region sizes. However, the size of coherent-scattering region and the size of particle are not equal exactly because of bound of crystal can have an amorphous cover.

When the lattice parameters of identify phases and standard parameters are considering some differences can be found. These differences can be explained by a non-equilibrium and a high dynamism of the processes of synthesis and crystallization. Effects of these conditions are defect structures of obtained crystalline nanostructures with an intrinsic high level of inner tough microdistortions proportional to Δd/d and also high dispersibility of synthesized phases. Therefore, the obtained data of the XRD bear out the electron microscopy results about boron carbide existence in the synthesized powder.

Table 1. Data of the XRD analyses

Crystalline phase	Content, % mass.	Lattice parameter experiment/theory, Å		Δd/d·10^{-3}
		a	c	
B_4C SG: R -3m	97.0	5.605/ 5.618	12.097/ 12.099	1
SiC SG: F-43m	0.5	4.366/ 4.348	4.366/ 4.348	7.9
C (graphite) SG: P 6_3 m c	2.5	5.569/ 2.470	6.778/ 6.790	4.2

In this study, all date are obtained by usage of modern analysis methods of nanomaterial. XRD patterns confirmed the presence of B_4C phase in disparate diffraction planes. TEM images of synthesized nano-powder showed that the mean particle size was between 10 nm and 1 μm. It allows making a conclusion of the possibility of the dynamic synthesis of crystalline boron carbide phase with CMPA.

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