

# Structure and properties of boron carbide ceramics fabricated by hot pressing

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**Abstract.** The hot-pressing production technique of high-density ceramics on polydisperse boron carbide base with an average particle size of  $2.1\mu\text{m}$  was studied. In some cases, a highly dispersed powder of chromium carbide was used as a sintering additive. Features of the structure were analyzed. The presence of pores between the grains was identified. Morphology of grains characteristic of highly dispersed boron carbide is observed.

## 1. Introduction

Boron carbide ( $\text{B}_{12}\text{C}_3$  or  $\text{B}_4\text{C}$ ) shows promise as the material with the high melting point, hardness, specific strength and chemical stability in aggressive media [1]. The coatings containing  $\text{B}_4\text{C}$  are widely used and investigated [2, 3]. However its use in manufacture is limited by the complexities of high-density ceramics preparation.

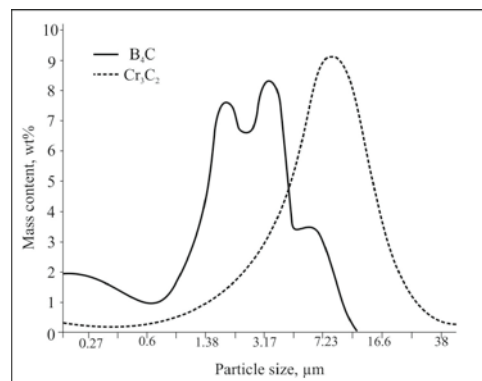
The production technique of high-density ceramics on boron carbide base could be improved via sintering additives. Refractory oxygen-free compounds could find application as the sintering additives, such as chromium diboride ( $\text{CrB}_2$ ) [4], titanium diboride ( $\text{TiB}_2$ ) [5], chromium carbide ( $\text{Cr}_3\text{C}_2$ ) [6]. Reaction sintering was carried out for an hour under an argon atmosphere at different temperatures [6]. The density of sintered products tended to grow with the increase of chromium carbide content and the temperature of sintering. The sample density reached 95% from the theoretical value at  $2150^\circ\text{C}$  and  $\text{Cr}_3\text{C}_2$  content in mixture of 25 wt%. The research objective is to study the sintering of polydisperse boron carbide by hot pressing and to examine some properties of the obtained ceramics.

## 2. Materials and experimental procedure

In some cases, a powder of boron carbide with the purity of  $\sim 98.5\%$  [7] and a sintering additive of  $\text{Cr}_3\text{C}_2$  powder with  $\sim 98.0\%$  purity [8] were used as starting materials. Particle size distribution of boron and chromium carbides are shown in figure 1.

In compliance with the procedure described in [9], the average particle sizes, standard deviations and asymmetry indicators (skewness) were determined by means of the «geometric» method. The data are presented below in the table 1.





**Figure 1.** Particle size distribution of boron carbide and chromium carbide samples.

**Table 1.** The results of sedimentation analysis.

Sample	The average size of particles, $\mu\text{m}$	Standard deviation	Skewness
boron carbide	2.1	2.47	-0.20
chromium carbide	7.1	2.10	-0.18

The values of the standard deviation are indicative of a wide range of particle size distribution (the particles are “poorly sorted” or polydisperse). We consider that it could have a positive effect on the process of high-density ceramics fabrication. Low values of the asymmetry indicator (skewness) turn out to be the proof of the histograms symmetry.

To determine the values of bending and compressive strength, the work-pieces from the powder of boron carbide were exposed to hot pressing at the temperature of 2100 °C, under pressure of 35MPa, and 45minute-holding time. Volumetric porosity and water absorption capacity were also estimated for the samples obtained. The work-pieces were made from the samples in order to define the material mechanical characteristics (i.e. bending strength and compressive strength). Bending /compressive strength tests were carried out with the use of Instron machines 3369.

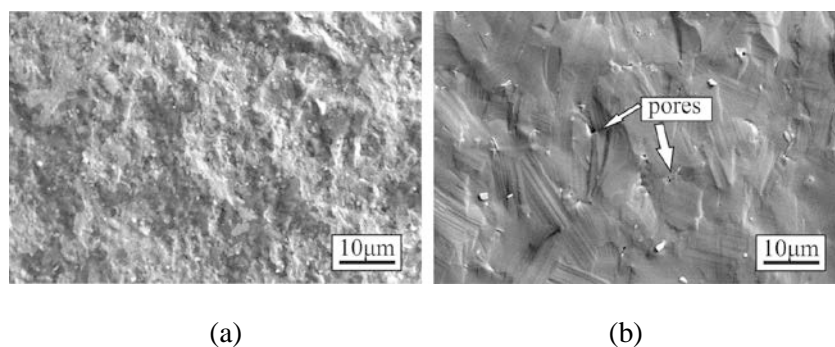
The other series of the tests was done to estimate density and microhardness of hot-pressed ceramics. Its microstructure was also examined. For comparison, we exposed to sintering not only boron carbide, but the mixtures with chromium carbide additives of 6 wt% and 20 wt%. Hot pressing was conducted in the argon flow, under pressure of 35MPa, at ~1950 °C. The temperature was set when taking into consideration the fact that boron carbide starts melting at this heat level [1] and, consequently it results in liquid-phase sintering. Microstructure studies of cleavage faces and polished sections were conducted with the use of CarlZeiss EVO50 scanning electron microscope (SEM). Microhardness was further determined on the polished sections. Microhardness was measured T with the use of 402 MVD (WolpertGroup) semiautomatic hardness meter.

### 3. Results and discussion

The values of volumetric porosity (0.01-0.02 %) and water absorption capacity (0.01 %) were extremely low for the sintered samples. The averaged values of bending and compressive strength were equal to 406 MPa and 1553 MPa for the samples under test, respectively. These averaged values are rather high, because the bending strength of boron carbide amounted to 340 MPa, and the compressive strength was in the range of 1100...1660 MPa.

While being sintered, boron carbide samples were found to shrink at ~ 1700 °C, but shrinkage of the samples with a sintering additive was observed at ~ 1500 °C. It's important to note that in accordance with [4], carbon oxide densification started at the temperature of 1800-1830 °C, but this process started at 1750 °C with the use of 10 wt%  $\text{Cr}_3\text{C}_2$  sintering additive. Polydispersity (i.e. wide-

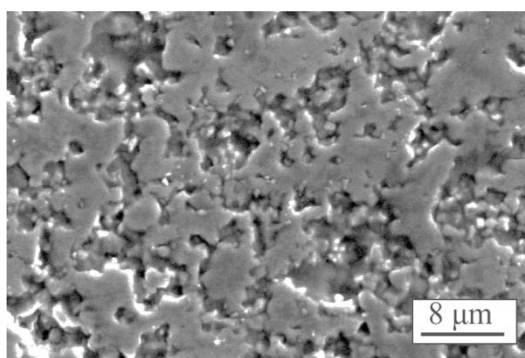
range particle size distribution) and high dispersiveness of carbide powders are certain to be the reason of low-temperature shrinkage start. The values of the samples density ratio were estimated in percent (%): № 1 – 95.0; № 2 – 96.0; № 3 – 96.5. Consequently, the density ratio is slightly affected by the use of a sintering additive. Micrographs of samples with cleavage surfaces are shown in figure 2.



**Figure 2.** Failure surface B<sub>4</sub>C (a) and B<sub>4</sub>C+6% Cr<sub>3</sub>C<sub>2</sub> (b).

The analysis of fractured cleavage faces gives evidence of dense structure formation and the lack of large pores and discontinuities. Minor quantities of closed pores are found in the samples containing boron carbide. The pores or voids have a spherical shape and their volume concentration is quite small, therefore they don't have any negative impact on the material properties. Volumetric porosity tends to decrease considerably with the increase of carbon carbide content. A cast structure is also observed owing to the addition of alloying elements at the time of sintering. Grain boundaries are practically invisible.

Sample structure analysis allowed us to determine the grain size of sample №3 containing 20 wt% of chromium carbide. Its grain size equals 0.5 μm (figure 3). The grain size for the rest samples is nearly the same.



**Figure 3.** The structure of B<sub>4</sub>C+ 20 % Cr<sub>3</sub>C<sub>2</sub>.

Microhardness reached the values of  $41 \pm 3.2$  GPa for sample №1, and  $46 \pm 2.8$  GPa for sample №2, and  $45 \pm 2.4$  GPa for sample № 3. The obtained results illustrate that the use of a sintering additive (in this case polydisperse chromium carbide) results in the increase of the sample microhardness. The microhardness values are comparatively high. They fall in the range of 29.7...47.9 GPa for the samples from boron carbide [1].

#### 4. Conclusion

The article presents the study of the hot-pressing technique applied for polydisperse boron carbide and for chromium carbide used as a sintering additive. Some characteristics of the obtained ceramics are

also reviewed. At relatively low pressure of hot-pressing (~ 35 MPa), the samples with the following quality attributes have been fabricated: volumetric porosity and water absorption capacity – not more than 0.02 %; the average value of bending strength – 406 MPa; the average value of compressive strength – 1553 MPa; relative density – 95-96%, microhardness – 42 GPa. The use of chromium carbide sintering additive resulted in the increase of microhardness up to 45-46 GPa.

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