Materials based on boron carbide obtained by reaction sintering

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Abstract. Porous boron-carbide billets containing different amounts of carbon (0-15 wt%) were infiltrated with molten silicon to produce dense materials (ρ = 99.6 % of theoretical) from starting B₄C powders with d₀.₅ = 90 and 12.5 μm. The phase composition, microstructure and mechanical properties of the material were studied. A solid solution of boroncarbide in silicon was determined to be present.

1. Introduction
Ceramics from of boron carbide possesses a low density, high hardness and Young's modulus, which allows the use of these materials in extreme conditions of high dynamic loads [1, 2]. However, the covalent bond in boron carbide creates difficulties in the preparation of dense materials at its base. Typically, these materials are prepared by hot pressing [3].

An alternative is the method of reaction sintering B₄C (RBBC). According to this, molded a preforms of boron carbide and the soot, which were impregnated with a silicon melt. During infiltration by capillary action, the liquid fills the pores and silicon reacts with the carbon black to form secondary silicon carbide [4–6]. Also, silicon partially dissolves boron carbide to form a solid solution B₁₂(C,Si,B)₃ which is significantly embrittle the material and negatively affect its mechanical properties, particularly crack resistance coefficient. For decreasing the formation of solid solution can restricting contact of molten silicon during sintering to the surface of the workpiece based on boron carbide.

The aim work is to study new aspects for RBBC-material, consisting in the impregnation of porous materials through the channels of sacrificial porous workpieces, study of the structure, phase composition and physical properties of sintered materials.

2. Starting materials and methods
The original components used in the work - boron carbide d₀.₅ = 42,0 μm (B₄C₁g) and d₀.₅ = 2,5 μm (B₄Csm), technical carbon black «К–354» and silicon «KR00» milled to size d₀.₅ = 0,5–1,0 sm. The compositions of the materials were shown in Table 1.
Samples of the compositions №1 and №2 were prepared through impregnation sacrificial preform; №3 composition samples prepared according to standard techniques reaction sintering (with an excess of silicon during impregnation) and used for comparison of properties.

| № composition | Content of components in workpiece, wt. % | Density $\rho \pm 0.02$ (g/cm$^3$) | Porosity $P \pm 0.1$ (%) |
|---------------|------------------------------------------|---------------------------------|--------------------------|
|               | $B_4C_{60}$ | $B_4C_{25}$ | Carbon | Si |                  |                  |
| 1             | 60          | 25          | 15     | -  | 2.70             | 0.9              |
| 2             | 56          | 24          | 15     | 5  | 2.72             | 1.2              |
| 3             | 60          | 25          | 15     | -  | 2.55             | 6.4              |

3. Results of the study
The results of XRF sample 3 (Figure 1, c) confirm the presence of a phase of Si, $B_{12}(C,Si,B)_3$, $\beta$-SiC and a small amount of boron carbide. The material structure 1 shows the phase of silicon content due to minimal dosed (optimal) amount entering the silicon workpiece. For the same reason, fewer phase content $B_{12}(C,Si,B)_3$ (Figure 1, a). When introduced into the batch 5 wt. % Si (composition 2) of the sintered sample is also present silicon phase and the $\beta$-SiC (Figure 1b). The intensity of the peaks corresponding phase $B_{12}(C,Si,B)_3$ is reduced, the secondary phase silicon carbide - is increased.

![Figure 1. X-ray phase analysis of material structure 1 (a), composition 2 (b), composition 3 (c)](image)

The microstructure of the material 1 is characterized by the presence of large grains of boron carbide $d_{0.5} = 39.5$ μm (gray, Figure 2a), which proves small grains $B_4C$ dissolution in the silicon melt. Introduction of additional Si in the composition of the material increases the solubility of grains $B_4C$ (Figure 2b). In the field of weak contact with the silicon melt intergranular porosity is observed (Figure 2c).

The obtained SEM photograph RBBC microstructure (Figure 3) demonstrates the presence of $B_4C$, $B_{12}(C,Si,B)_3$, $SiC^{II}$ ($\beta$-SiC), Si phases in the materials, which confirms the conclusions of the microstructure analysis made on an optical microscope (Figure 2).

Properties reactive sintered materials are shown in Table 2.

Dense materials (composition 1) have a high level of mechanical performance, far exceeding the level of properties RBBC-materials sintered under standard reaction sintering conditions (composition 3, Table 2).

When impregnate porous workpieces with silicon melt through the pores of sacrificial specimens, materials based on boron carbide with a high density ($\rho \approx 99.0$ %), which have a high level of mechanical characteristics, are obtained, compared to materials obtained by standard impregnation with an excess amount of silicon [4].
Figure 2. The microstructure of materials of structure 1 (a), composition 2 (b), composition 3 (c)

Figure 3. The microstructure of the material composition 3

Table 2. Mechanical properties RBBC-materials

| №  | composition | Elastic modulus $E_{clast} \pm 10$ (GPa) | Flexural strength $\sigma_f \pm 10$ (MPa) | Fracture toughness $K_{IC} \pm 0.2$ (MPa·m$^{1/2}$) | Vickers hardness $HV \pm 0.1$ (GPa) |
|----|-------------|----------------------------------------|----------------------------------------|----------------------------------------|----------------------------------------|
| 1  | 360         | 450                                    | 4.8                                    | 30.5                                   |
| 2  | 340         | 420                                    | 4.2                                    | 28.5                                   |
| 3  | 320         | 380                                    | 3.8                                    | 27.0                                   |

From the comparative analysis based on boron and silicon carbide materials properties shows that obtained a reaction-sintered B$_4$C, the level of physico-mechanical characteristics superior reaction-sintered SiC and approaches the level of properties of hot-pressed materials based B$_4$C (Table 3).
### Table 3. Comparative characteristics of materials

| Material                        | ρ (g/cm³) | P (%) | σₚ (MPa) | $K_{IC} = 0.2$ (MPa·m$^{1/2}$) | HV (GPa) |
|---------------------------------|-----------|-------|----------|-------------------------------|----------|
| Reaction-sintered B₄C          | 2.70      | 0.9   | 450      | 4.8                           | 30.5     |
| A hot-pressed composite B₄C-Al [7] | 1.67      | 28.0  | 125      | -                             | -        |
| Hot-pressed B₄C [3]            | 2.54      | 1.0   | 665      | 5.3                           | 36.1     |
| Reaction-sintered SiC [4]      | 3.10      | 0.5   | 400      | 4.5                           | 21.0     |

### References

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