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STRUCTURE, PHYSICOMECHANICAL PROPERTIES, AND SPECIAL FEATURES OF FAILURE OF HOT-PRESSED BORON CARBIDE BASE CERAMICS

O. N. Grigor'ev, V. V. Koval'chuk, V. V. Zametailo, R. G. Timchenko, D. A. Kotlyar, and V. P. Yaroshenko

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In recent years nonoxidic ceramics with covalent bond, based on silicon carbide, silicon nitride, aluminum nitride, have found application in various branches of new technology. A characteristic feature of these compounds are good physicomechanical and operational properties. This group also includes materials based on boron carbide which have high values of hardness and of elastic constants combined with low specific weight. However, single-phase materials based on boron carbide have insufficient strength and crack resistance [1], and that limits their application in tools and structures operating under intense mechanical and thermochemical loads.

An effective way of increasing the strength and crack resistance of ceramics is the devising of heterophase structures by adding to the matrix brittle, tough, or fibrous components of a second phase. It was, e.g., discovered that the physicomechanical characteristics of materials were substantially improved in the systems Si_3N_4–TiC, SiC–TiC, SiC–TiB_2, B_4C TiB_2, and B_4C–ZrB_2 [2-7]. One or simultaneously several mechanisms of increasing fracture toughness are realized in them [7, 8]: deflection of the path of crack propagation from rectilinearity, microcracking around particles of the second phase, ramification of

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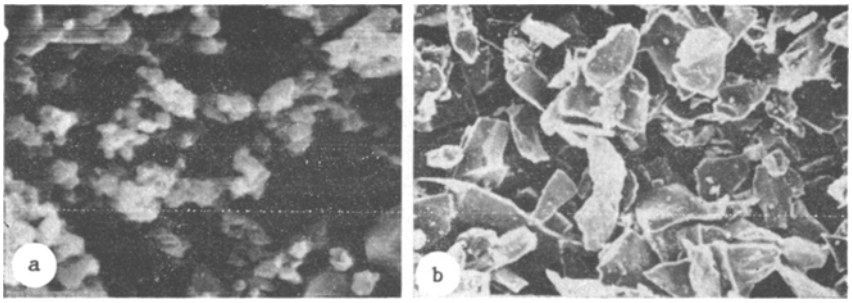

Fig. 1. Microstructure of the initial powders of boron carbide obtained by synthesis of the elements (a) and by carbothermal reduction (b). Magnification: a) 5000; b) 1000.

TABLE 1. Characteristics of the Initial Powders

Powder	Chemical composition, wt. %				Grain size, μm			Specific surface, m^2/g
	B_{tot}	B_2O_3	C_{tot}	Fe	fine	coarse	medium	
Synthesized from elements (TU 6-09-668-76)	78,8	0,83	21,1	0,07	0,5	4	1,0	6,75
Technical boron carbide (GOST 5744-74)	71,0	0,3	28,2	0,21	1,5	30	16	3,22

the crack. Changes in the nature of crack propagation are due to the fact that the front of crack propagation interacts with the fields of residual stresses arising in the material on account of the difference between the coefficients of thermal expansion (CTE) and of the modulus of elasticity of the matrix and particles of the second phase. The specified level of internal stresses in boron carbide can be ensured by SiC, TiB_2, and ZrB_2. The mean values of the CTE of boron carbide and silicon carbide practically coincide, and the internal stresses in the material are due to the anisotropy of the CTE of B_4C and SiC, whereas the CTE of TiB_2 and ZrB_2 is substantially larger than that of B_4C.

In the present work we investigate the structure, the physicomechanical properties, and the pecularities of failure of the single-phase boron carbide, and also of materials based on it: B_4C–SiC, B_4C–TiB_2, B_4C–ZrB_2. The initial materials were boron carbide powders obtained by synthesis from the elements and by carbothermal reduction (Fig. 1, Table 1). To obtain material based on boron carbide, we added silicon, titanium, zirconium oxides to the charge, and to ensure completion of the chemical reaction, we also added free carbon in the form of soot.

It was established that in the process of reaction sintering under pressure, the interaction between boron carbide, oxides, and oxygen results in the formation of ceramic material with the phase composition B_4C–SiC, B_4C–TiB_2, B_4C–ZrB_2. There are also minute amounts of other phases present (FeB, SiB_6), which has to do with the pollution of the powders in production.

We investigated the grain size distribution of the initial powders on instruments "Culter TA-2" and "Acusorb-210," and with their aid we also determined the specific surface. The test specimens were obtained by the method of hot pressing on an installation with induction heating in graphite molds with the use of a protective coating based on BN. The bending strength of the specimens was determined on a tensile testing machine R-0.5. The microstructure was studied with the aid of optical and scanning electron microscopy. Crack resistance was determined under bending load imposed on notched beams (width of the notch was 0.12 mm). The tests were carried out on a universal testing machine 1958U10 (speed of the beam 1 mm/min). In addition to that, we determined crack resistance K_{1C} by microindentation according to the method of [9]. Vickers hardness was measured under a load of 10 N on an instrument TP-7R-1 which was also used for crack initiation by pressing a diamond Vickers pyramid into the surface of the microsection under a load of 100 N. The modulus of elasticity was evaluated by the speed of an ultrasound signal passing through the specimen [10].

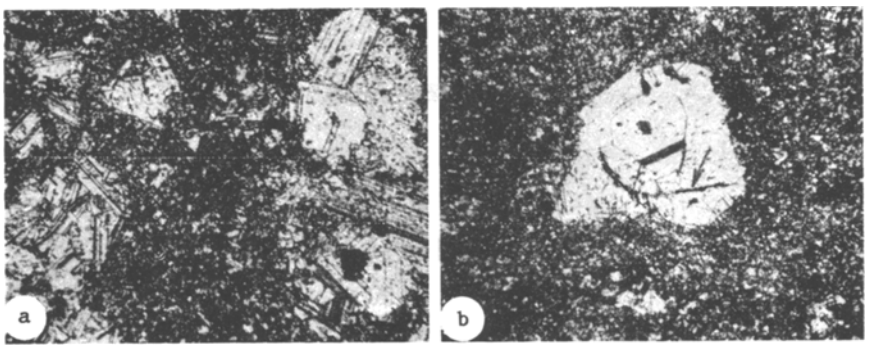

Fig. 2. Microstructure of boron carbide specimens: a) sections of anomalous grain growth; b) microcracks (marked by arrow) in coarse grains. ×120.

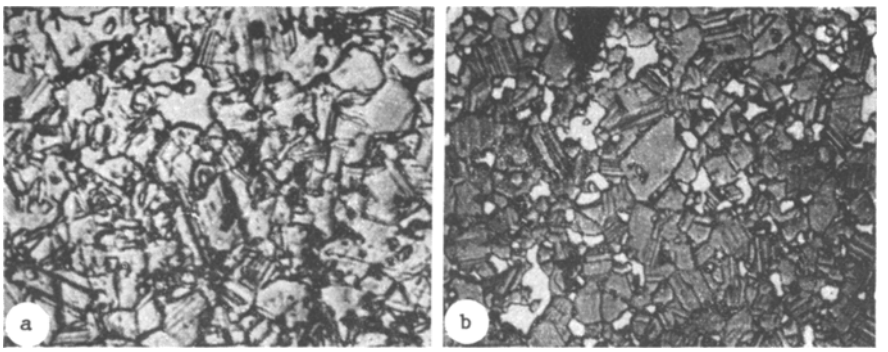

Fig. 3. Microstructure of the materials B_4C – 10% SiC (a) and B_4C – 15% ZrB_2 (b). ×800.

Single-phase hot-pressed boron carbide has an inhomogeneous structure because of nonuniform grain growth in the process of accumulative recrystallization. In many cases there arise conditions of their anomalous growth [11, 12], and consequently the size of some grains exceed 300 μm (Fig. 2). Owing to residual stresses arising in consequence of anisotropy of the CTE and of the modulus of elasticity of boron carbide, spontaneous microcracking is possible, and this was also experimentally discovered (Fig. 2b). Single-phase hot-pressed boron carbide has comparatively low crack resistance (K_{1C} = 3.0-3.6 MPa·$m^{1/2}$) and strength (σ_{ben} = 300-360 MPa). It was discovered that the strength of material with inhomogeneous structure containing large grains may be as low as 200 MPa.

Addition of oxides to the charge greatly increases the speed of shrinkage; this makes it possible to obtain material with low porosity (~2-3%) at lower temperatures and with brief isothermal holding. The processes of accumulative recrystallization and of anomalous grain growth are thereby slowed down, and this promotes the formation of ceramic materials with homogeneous, comparatively fine-grained structure (4-6 μm) and good physicomechanical properties (Fig. 3).

Failure of the specimens is of a mixed nature: intercrystalline and transcrystalline. On sections of intercrystalline failure of heterophase materials we find grain-boundary interlayers formed in the process of liquid-phase sintering and recrystallization (Fig. 4). The transition from transcrystalline to intercrystalline failure occurs at some critical grain size $d_{cr} \approx 3$ μm, which is the same for single-phase and heterophase materials. However, in heterophase materials this transition occurs within a wider range of grain sizes (Fig. 5).

An analysis shows that the critical grain size is determined by the strength of the grain boundaries. That the mean critical grain size of single-phase as well as heterophase materials is constant is due to the fact that they have equal strength of the grain boundaries. On the other hand, the broad range of grain sizes within which we find a transition from intercrystalline to transcrystalline failure in heterophase materials

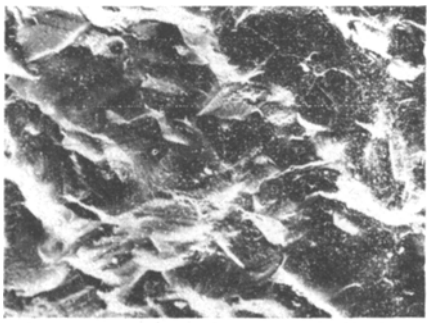

Fig. 4

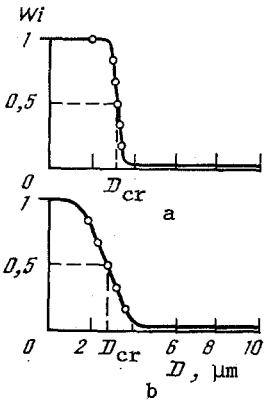

Fig. 5

Fig. 4. Fracture surface of material of the system B_4C–SiC. ×2000.

Fig. 5. Dependence of the proportion of intercrystalline fracture of boron carbide (a) and of the material B_4C – 10% SiC (b) on the grain size.

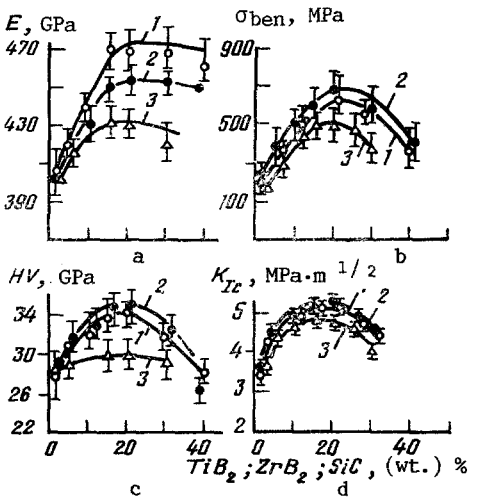

Fig. 6. Change of the modulus of elasticity (a), of the bending strength (b), of hardness (c), and of crack resistance (d) of ceramic materials in dependence on the content of second phase in them: 1) TiB_2; 2) ZrB_2; 3) SiC.

indicates great dispersion of the strength of grain boundaries, a considerable proportion not only of strong (in comparison with the mean) but also of weakened boundaries. The great dispersion of strength of the grain boundaries of heterophase materials may be a consequence of fields of nonuniform interphase internal stresses as well as of the existence of grain-boundary interlayers differing from each other in composition, structural state, and strength.

Under conditions of considerable dispersion of strength of the grain boundaries of heterophase materials, external loading may induce processes of microcracking. In that case microcracks forming along "weak" boundaries are efficiently inhibited in the region of strong boundaries, and this, in the final analysis, leads to increased crack resistance of heterophase material and also, as shown by the investigations, to higher strength and hardness (Fig. 6). Besides that, increased crack resistance may be partly due to processes of deflection of the cracks from rectilinear propagation (Fig. 7).

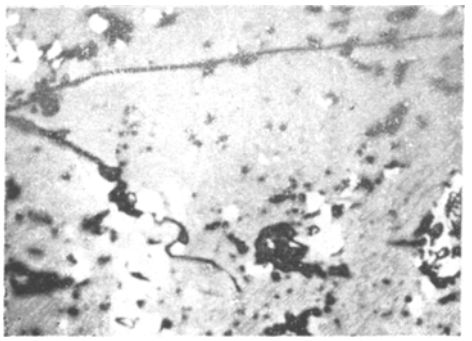

Fig. 7. Propagation of a radial crack from the indent in material of the system B_4C–TiB_2. ×1000.

The mechanical properties of heterophase materials improve considerably when the content of the second phase amounts to ~15%. A further increase of its concentration impairs crack resistance and other characteristics because the internal stresses then exceed the optimal level.

Thus an addition of SiO_2, TiO_2, and ZrO_2 to the charge helps obtain highly dense heterophase materials based on boron carbide. Investigation of additives that activate sintering makes it possible to make effective use of technical boron carbide powders for obtaining ceramic materials of the systems B_4C–SiC, B_4C–TiB_2, and B_4C–ZrB_2 with high stable level of physicomechanical properties.

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