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REGULARITIES OF THE SYNTHESIS OF THE TiB_2-Al_2O_3 COMPOSITE CERAMIC POWDER AND THE MATERIAL BASED ON IT

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With a certain selection of phases, composite ceramic materials become insensitive to stress concentration and crack formation and propagation, and resist alternating and prolonged loads well. A knowledge of the behavior of each component phase of the composition and of their interplay permits the production of materials with preset mechanical properties, such as strength, rigidity, heat resistance, and chemical stability.

The present study investigates the regularities of the synthesis of the TiB_2-Al_2O_3 composite ceramic powder, which is a disordered mixture of TiB_2 in Al_2O_3. We also investigated the physicomechanical characteristics of the material produced by hot pressing of this powder.

The combustion of the $TiO_2+3B_2O_3+10Al$ system was investigated in a constant-pressure bomb. Powders with the following characteristics were employed: boron oxide B_2O_3 - a content of the basic substance of 98.5% and a particle size smaller than $500 \cdot 10^{-6}$ m; aluminium with a particle size ranging from $5 \cdot 10^{-6}$ to $375 \cdot 10^{-6}$ m; and titanium oxide with a specific surface of $0.75 \cdot 10^3$ m^2/kg.

From premixed powders, cylindrical samples were pressed, which were ignited from the end by a Nichrome spiral and burned in constant-pressure bomb [1] in gaseous argon. The combustion rate and temperature were determined as an average of 3-4 tests using a VR 5/20 tungsten-rhenium thermocouple [2] of thickness $(100\text{-}200) \cdot 10^{-6}$ m.

We performed chemical, x-ray phase, metallographic, and local x-ray spectrum analyses of the product obtained. The change in the sample length was estimated from the equation

$$\Delta h = \frac{h_{\text{in}} - h_{\text{f}}}{h_{\text{in}}} \cdot 100\%,$$

where h_{in} is the initial dimension of the sample and h_{f} is the dimension of the sample after combustion.

The wear resistance of compact samples produced from 29% TiB_2 - 71% Al_2O_3 powder was studied using a "NEKKER" unit; high-temperature hardness was also ascertained (the Vickers method, σ_{bend}, Rockwell hardness HRA).

With increasing pressure, the combustion rate in some systems rises [3], whereas for others it is independent of pressure [4] or is of anomalous character [5].

Figure 1 shows the parameters of combustion and the phase composition as functions of the pressure of an inert gas. The combustion rate decreases by 1.4 times and the combustion temperature (T_c = 2100-2380°C) and the sample shrinkage (Δh = 0-19%) increase as the pressure rises from 0.1 to 3 MPa, remaining constant afterwards. X-ray phase analysis revealed a two-phase product throughout the pressure range. Aluminum oxide has a microhardness of 21,320 MN/m^2-24,500 MN/m^2, and titanium diboride has a microhardness of 29,260 MN/m^2-38,900 MN/m^2; moreover, chemical analysis demonstrated the presence of boron oxide, which corresponds to low values of microhardness.

The combustion temperature over the entire pressure range is in excess not only of the melting temperatures of boron oxide and aluminum but also of the melting temperature of aluminum oxide, and, furthermore, the final product has a eutectic at 1800°C. The indicated factors cause the samples to shrink.

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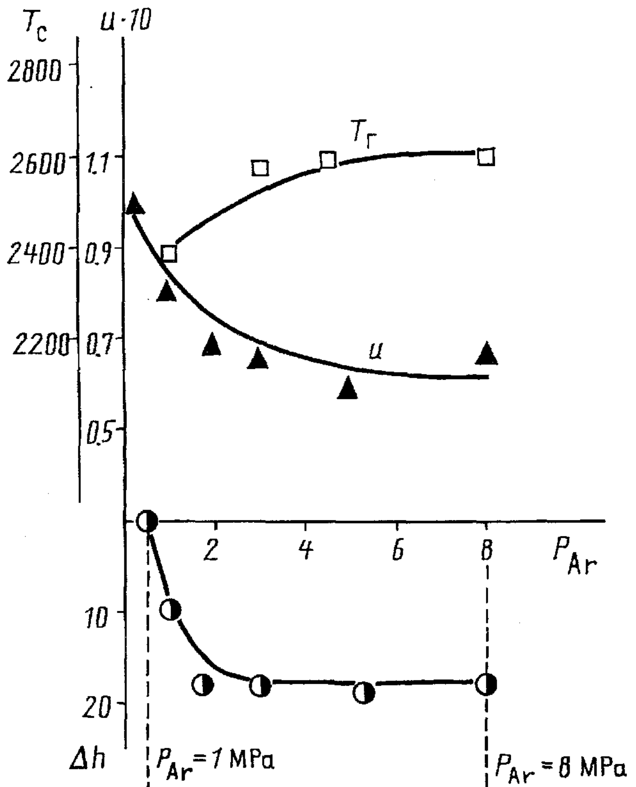

71% Al_2O_3-29% TiB_2	
Al_2O_3	TiB_2
21440	28260
16820	38900
24500	
21320	

71% Al_2O_3-29% TiB_2	
Al_2O_3	TiB_2
26280	29260
14260	46720
21320	
18920	

Fig. 1. Combustion temperature T, °C, combustion rate u, m^2/sec, sample shrinkage Δh, %, and microhardness, MN/m^2, as functions of the argon pressure P_{Ar}, MPa; $\rho = 1.53$ g/cm^3, $B_2O_3 < 500$ μm, Al = 5 μm, $d_s = 25$ mm.

Thermodynamic analysis at the combustion temperature indicated a gas phase consisting of B_2O_2 and B_2O_3; the maximum pressure at the combustion temperature is 1 MPa. An increase in pressure reduces the gas generation, thus leading to an increase in sample shrinkage, and the heat transfer by adsorbed and forming gases from the combustion front to the initial mixture, thereby decreasing the combustion rate. Mass-spectroscopic analysis of the gas phase after the combustion indicated 94.7% hydrogen. Apparently, it is released from boron oxide (an impurity gas release) because, during blending of the original mixture, moisture can be absorbed from the air due to hygroscopicity.

Heat loss has a great impact on the propagation of the combustion wave. Study [6] found that the combustion rate rises as the plate thickness decreases. This points to the secondary role of the heat loss and to the important role of the impurity gas release.

An increase in the sample diameter from 0.01 to 0.04 m (Fig. 2) decreases the combustion rate by 1.6 times, increases the sample shrinkage by 1.4 times, and lowers the combustion temperature from $T_c = 2510$ to 2400°C. Chemical analysis of the final product demonstrated that, with a sample diameter of $1.5 \cdot 10^{-2}$ m, the content of unreacted aluminum is 1.8 times, and that of boron oxide is 1.7 times, as great as that at $d_s = 4 \cdot 10^{-2}$ m.

The decrease in the combustion rate is connected with melting of the final product at the combustion temperature and its penetration into the combustion zone, where aluminum and boron oxide are in the liquid state. This sets up diffusional hindrances to the reacting components. With small-diameter samples, there is rapid cooling

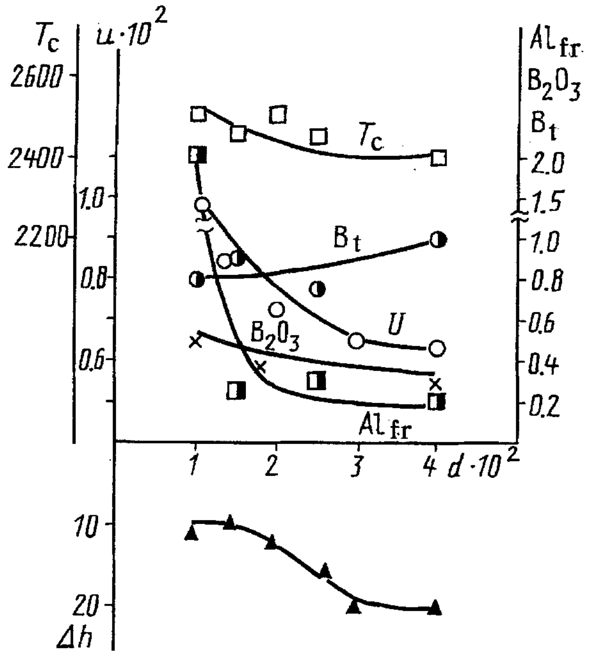

Fig. 2. Combustion temperature T_c, combustion rate u, sample shrinkage Δh, and content of total boron B_t, free aluminum Al_{fr}, and boron oxide B_2O_3, %, in the final product as functions of the sample diameter, m; $\rho = 1.58$ g/cm^3, $P_{Ar} = 30$ atm, $B_2O_3 < 500\,\mu$m.

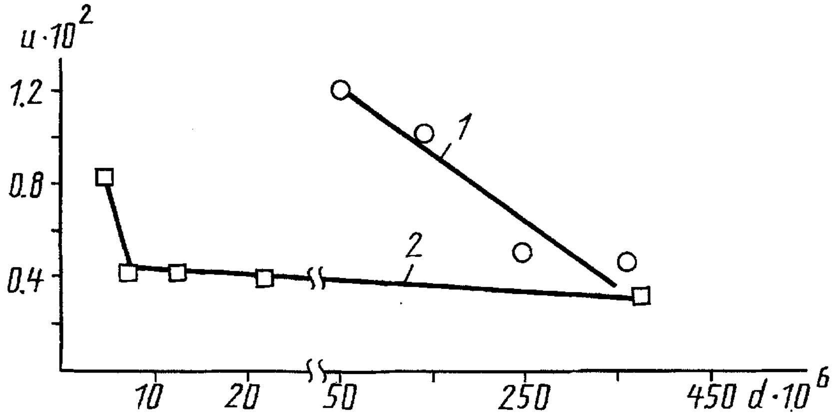

Fig. 3. Combustion rate as a function of dispersity of boron oxide (1) ($\rho = 1.60$ g/cm^3, $d_s = 25$ mm, $P_{Ar} = 30$ atm, and Al = 5 μm) and of aluminum (2) ($\rho = 1.64$ g/cm^3, $d_s = 25$ mm, $P_{Ar} = 30$ atm, $B_2O_3 < 500\,\mu$m).

of the product melt and the shrinkage is less due to heat removal. Hence, the effect of the product melt diminishes, which causes the combustion rate to rise.

The decrease in the combustion rate from $1.22 \cdot 10^{-2}$ to $0.33 \cdot 10$ m/sec as the dispersity of the boron oxide increases from $50 \cdot 10^{-6}$ to $358 \cdot 10^{-6}$ m (Fig. 3)and from $0.77 \cdot 10^{-2}$ to $0.21 \cdot 10^{-2}$ m/sec as the dispersity of aluminum increases from $5 \cdot 10^{-6}$ to $375 \cdot 10^{-6}$ m is related to a change in the characteristic time of component mixing in the heating and reaction zones. At small d_{part}, the components are completely heated and intermix, and the combustion characteristics and the rate of heat release are determined by a rate of the chemical reaction, thus representing a kinetic mode. The time of movement of the heating front is $\tau = a/u^2$, and the time of particle heating is $\tau = d^2/a$ [7]. As d_{part} increases, the heating time increases, mixing in the heating zone deteriorates, and the rate of heat release is determined by the rate of reagent diffusion to the reaction surface, which is a diffusion mode. Here the combustion rate falls, and the quality of the final product worsens. The combustion rate is $u \sim 1/d_{part}$ [1].

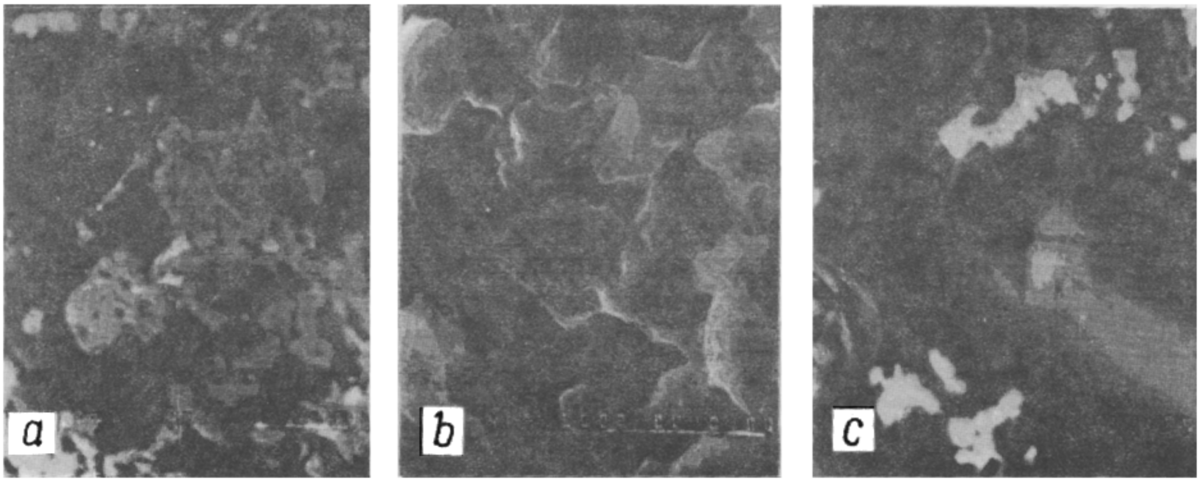

Fig. 4. Microphotography of the 29% Ti-B_2 - 71% Al_2O_3 compact material in COMPO filming mode: a) general view; light part indicates, TiB_7 and dark part, Al_2O_3. ×3000; b) etching in the KOH + HNO_3 melt at 800°C over 1 min, aluminum oxide grains, ×4000; c) indexer imprint in the Al_2O_3 phase, ×2200.

The investigations performed enabled us to obtain a composite 29% TiB_2 - 71% Al_2O_3 ceramic powder with a minimum content of impurities. By hot pressing of this powder at a pressing temperature of $T_{pr} = 1850^oC$ and a pressing pressure of $P_{pr} = 1.9$ MPa, we obtained a compact material, whose microstructure is shown in Fig. 4a. The sample has a relative density of $\rho_{rel} = 99.1\%$, $\sigma_{bend} = 1074$ MN/m^2, a Young's modulus of 814 kN/mm^2, a wear of $0.01316 \cdot 10^3$ kg/m^3, a Rockwell hardness HRA = 91.5, and a Vickers hardness HV= 11,790 MN/m^2 at T = 20°C and HV = 3040 MN/m^2 at T = 900°C, which greatly exceeds the strength characteristics of the constituent phases [8, 9]. The analysis revealed that the interphase coupling is purely mechanical.

The increase in the strength characteristics is caused by the disruption of continuity of the aluminum oxide phase by the titanium diboride phase. A microcrack formed, for instance, in the aluminum oxide phase changes its direction on entering the titanium diboride phase (Fig. 4c). The propagation of the main cracks which result in complete destruction of the material, is hindered. Figure 4b shows the structure of the etched material. The titanium diboride phase is dissolved. The arrangement of aluminum oxide grains is clear. We can envision how many times the main crack changes its direction up to a complete destruction of the material.

The material produced from the powder synthesized using SHS surpasses in physicomechanical properties the material studied in [10].

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