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PRODUCTION OF BORIDE-CONTAINING CERAMIC MATERIALS FROM BORATE ORES BY THE SHS METHOD

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The possibility is shown for producing boride-containing ceramic materials with the use of borate ores. Optimum conditions for SHS in the system of borate ore-titanium oxide-aluminum are chosen.

At present, there is a clear trend toward the development of works on creation of solid ceramic materials using borides for instrumental and structural ceramics. Works on production of ceramics in the TiB_2-Al_2O_3 system are known [1]. In this case the initial substances should be highly pure. Furthermore, large energy expenditures (1900°C) are required for sintering the initial substances.

The aim of this work is to obtain ceramic materials containing titanium borides and aluminum oxide by the SHS method directly from borate ore as well as from wastes that contain borogypsum.

The borate ore of the Indersk deposit had the following composition (wt.%): B_2O_3 20; CaO 27; MgO 9.27; SiO_2 5.8; SO_3 24.7; F 0.2. The phase composition is represented mainly by the minerals ionite $2CaO\cdot 3B_2O_3\cdot 13H_2O$ and gypsum (about 40%); ascharite, hydroboracite and quartz are present as impurities.

We performed the calculation of the charge composition and the amount of the necessary reducing agent (aluminum) on the basis of the reaction

$$10Al+3B_2O_3+3TiO_2=3TiB_2+5Al_2O_3.$$

In addition, the amount of aluminum necessary for reduction of $CaSO_4$ to sulfides was taken into account.

Thus, in order that the reactions be conducted completely, the charge composition must include ore, aluminum, and titanium oxide. The samples were obtained in the following manner: the charge was pressed into a billet under the different pressures, was thermostated in a furnace, and then was fired by magnesium. However, we failed to initiate the SHS reaction in the samples of the above-mentioned initial composition, because melting aluminum filled the reaction space. Therefore, aluminum oxide was introduced into the charge as a diluent.

To choose the optimal conditions for the process, we varied the composition, the pressing pressure, the temperature of thermostatic control, and the amount of inert diluent. It was established that an increase in the pressing pressure to 70-100 kg/cm^2 led to the breaking of the sample during combustion, and therefore the pressure was reduced to 40-50 kg/cm^2. The temperature from 600 to 700°C was optimal for thermostating. At this temperature the samples easily ignited from the burning magnesium. It is known that just at this temperature the majority of borates are subjected to so-called borate regrouping, during which a considerable quantity of energy is generated. The sharp convergence of the crystalline lattice elements provides the basis for this process [2]. Thus, the ore itself under heating gives an exothermal effect, which favors the appearance of the SHS reaction. The SHS reaction is accompanied by substantial gas liberation of sulfur oxides, as a result of which the obtained sample is very porous. The optimal compositions, not altering the sample shapes after SHS, contain 37.4%; in such compositions the reaction of titanium diboride formation is accomplished to the utmost. The mass loss in combustion corresponds to liberation of gaseous sulfur oxides. Thus, the sample of the composition that is optimal for the SHS reaction gives a mass loss of 8.7%. The content of SO_3 in the initial sample is also about 8%. A very small amount of sulfides remains in the solid product.

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The determination of the boron content in the initial sample and in the SHS products shows that there is virtually no loss of B_2O_3 in the course of combustion. The absence of titanium oxides in the burnt sample was established by selective dissolution.

According to RPA data, in the burnt sample there are titanium diboride, α-Al_2O_3, CaS, and two spinels of uncertain composition with cell parameters of 8.011 and 8.057 Å.

In connection with the fact that the samples after combustion became very porous and contained a certain amount of sulfides, we attempted to obtain from them the more inert and dense materials. For this purpose we ground up the samples, washed off traces of sulfides from them, flushed out and dried them, and then again pressed them into billets and annealed them at a temperature of 800-900°C; such compositions were readily sintered. In an hour of annealing we obtained dense solid samples (specific weight 2.42 g/cm^3) with high refractoriness. During the tests, the samples were heated by a high-temperature open flame (2400°C) with subsequent rapid cooling. After a 20-fold cycle no cracks appeared, and an insignificant mass loss (0.2%) was observed. Under length heating blue coloration occurred on the surface of the samples, presumably one of the titanium oxides, Ti_3O_5.

Thus, use of the SHS method allows us to obtain directly from the borate ores (omitting the stage of liberation of pure boric acid or boric anhydride) boride-containing products suitable for production of ceramic materials in the present case.

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