SELF-PROPAGATING HIGH-TEMPERATURE SYNTHESIS OF Si_3N_4-SiC USING FERROSILICIUM AND SHUNGITE*

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A new approach to the synthesis of silicon nitride powders by nitriding multicomponent reaction mixtures containing industrial iron-containing (ferrosilicon) and natural oxide materials (zircon, ilmenite) by the SHS method was developed at the Tomsk Scientific Center of SB RAS [1, 2]. The main advantage of the SHS method is minimal energy costs, since the synthesis is supported by the energy of chemical reactions. The method for obtaining composite powders is based on the silicon nitride reaction during the interaction of Si with nitrogen, which provides the main heat release during the propagation of the combustion reaction wave. This work is a continuation of our studies and is aimed at obtaining the Si_3N_4-SiC composition during the SHS- nitriding of ferrosilicon with the addition of natural oxide materials (schungite).

To estimate the maximum combustion temperature and the equilibrium composition of the products, a thermodynamic calculation of the synthesis of silicon nitride composites during the interaction of ferrosilicon with schungite additives in nitrogen was performed. The TERRA software package was used for calculations [3]. Calculations showed that with an increase in the amount of shungite, regardless of the nitrogen pressure, a slight decrease in the combustion temperature was observed; at adiabatic temperature, silicon nitride was in equilibrium with the dissociation products such as silicon and nitrogen. The composition of the reaction products is represented by the components as follows: Si_3N_4, SiC, Si, SiO_2, Fe, Fe_3C. In addition, SiO is formed, which is in the gas phase.

The mechanisms of the combustion of ferrosilicon in the presence of a natural material (schungite) was revealed for the synthesis of the Si_3N_4–SiC composition. It was shown that the addition of shungite (1–30 wt.%) to the starting "ferrosilicon – schungite" mixture led to a decrease in the nitriding degree of ferrosilicon. This is due to a decrease in the proportion of nitride-forming element in the starting mixture. An increase in the nitrogen pressure from 1.5 to 6 MPa led to an increase in the nitriding degree due to the increased supply of nitrogen to the reaction zone. The minimum sample diameter at which the combustion process takes place was 30 mm.

SHS nitriding of ferrosilicon with the addition of shungite was conducted in the surface combustion mode, as evidenced by the heterogeneous structure of the combustion product. The external layer of the burned samples is the unburned starting material in the form of a "crust". The thickness of the external layer of the sample decreases with an increase in the addition of 20-30% schungite. The macrostructure of the samples is homogeneous, has a light gray color with a greenish tint. It should be noted that an increase in the nitrogen pressure contributes to the decrease in the layer of the unburned starting material (i.e., the "crust" becomes thinner throughout the height of the sample). The phase composition of the synthesis products with the addition of schungite up to 15% both in the central part and in the external layers of the sample is represented by the phases as follows: β-Si_3N_4, SiC, Si_2N_2O, $FeSi_2$, FeSi и α-Fe. The presence of the initial alloy component such as the $FeSi_2$ phase in the combustion products indicates the incompleteness of the nitride formation process. The addition of shungite in an amount of 20% or more results in the formation of the phases as follows: β-Si_3N_4, SiC, Si_2N_2O, FeSi и α-Fe. Thus, it was shown that SHS nitriding of ferrosilicon powders with schungite additives makes it possible to obtain a Si_3N_4-SiC composition of different purity depending on the pressure N_2 and the composition of the starting mixture.

References

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