Combustion synthesis of highly dispersed powder composition AlN-SiC using sodium azide and polytetrafluoroethylene

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Highly dispersed (less than 1 micron) ceramic powders AlN and SiC are promising for the creation of new composite materials AlN-SiC with a complex of properties such as high strength, thermal stability, chemical resistance. The use of a simple energy-saving method of self-propagating high-temperature synthesis (SHS) of the AlN-SiC composition in the combustion mode of mixtures of inexpensive reagents attracts great attention of researchers [1]. The process of azide SHS, in which sodium azide powder NaN_3 as nitriding agent and halide salts are used, provides great opportunities to regulate the dispersion and structure of synthesized ceramic powders, bringing them to the nanoscale level. The application of azide SHS with halide salt $(NH_4)_2SiF_6$ and carbon black made it possible to synthesize highly dispersed AlN—SiC powder compositions with different ratios of nitride and carbide phases, however, the amount of SiC phase synthesized in experiments (up to 5.9 wt. %) turned out to be significantly lower than the expected theoretical amount (up to 79.6%) [1]. At the same time, combustion products contained a water-insoluble admixture of Na_3AlF_6 cryolite salt in noticeable amounts from 7.7 to 15.5%, as well as an admixture of silicon nitride (up to 2.2%) and unreacted free silicon (up to 0.9%).

In this regard, in this work, the use of polytetrafluoroethylene C_2F_4 in the azide SHS method as an initial carbidizing and activating reagent instead of carbon black and salt $(NH_4)_2SiF_6$ was investigated, which promoted the formation of silicon carbide in large quantities, and in the form of nanofibers and nanoparticles [2]. In this case, the initial reaction equations for the synthesis of target AlN–SiC compositions with a molar phase ratio from 1:4 to 4:1 had the following form:

$$2Al + 2Si + 4NaN_3 + C_2F_4 = 2AlN + 2SiC + 4NaF + 5N_2, \quad (1)$$

$$2Al + 4Si + 8NaN_3 + 2C_2F_4 = 2AlN + 4SiC + 8NaF + 11N_2, \quad (2)$$

$$2Al + 8Si + 16NaN_3 + 4C_2F_4 = 2AlN + 8SiC + 16NaF + 23N_2, \quad (3)$$

$$4Al + 2Si + 4NaN_3 + C_2F_4 = 4AlN + 2SiC + 4NaF + 4N_2, \quad (4)$$

$$8Al + 2Si + 4NaN_3 + C_2F_4 = 8AlN + 2SiC + 4NaF + 2N_2, \quad (5)$$

The results of thermodynamic calculations of these reactions are presented, according to which the adiabatic temperatures (2704-3216 K) are sufficient for the combustion regime, and the composition of the reaction products corresponds to the right parts of equations (1)-(5). The experimental study was carried out by burning the initial powder mixtures in bulk in a 4.5 L reactor in an atmosphere of gaseous nitrogen with a pressure of 4 MPa. The study showed that the synthesized compositions are agglomerates of powders from submicron particles ranging in size from 100 to 500 nm, the phase composition of which includes impurities of silicon nitride from 3 to 7%, cryolite salts from 4 to 15%, but the content of the SiC phase is large (from 11 to 50%) and much closer to the theoretical composition according to equations (1)–(5), in contrast to the results of [1].

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References

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