

## NITRIDING OF FERROSILICON – ILMENITE – ALUMINIUM MIXTURE IN COMBUSTION MODE

O.G. KRYUKOVA, K.A. BOLGARU, T.V. TATARINOVA

*Tomsk Scientific Center of the Siberian Branch of the Russian Academy of Sciences, Tomsk, Russia*

$\beta$ -SiAlON is formed in the Si-Al-O-N system. It is a solid solution of variable composition  $\text{Si}_{6-z}\text{Al}_z\text{O}_z\text{N}_{8-z}$  ( $z \sim 0-4,2$ ) formed on the basis of  $\beta$ - $\text{Si}_3\text{N}_4$  by substitution of Si atoms for Al and N atoms for O. SiAlON has unique performance characteristics (stability at high temperatures, high corrosion-, heat- and wear-resistance, high hardness and strength), which cause its application as high-temperature structural ceramics and corrosion-resistant materials [1]. When sialon is doped with rare-earth elements as phosphors in white LEDs [2].

Self-propagating high-temperature synthesis (SHS) is widely used to produce  $\beta$ -sialone [3, 4]. As a starting component for the synthesis of  $\beta$ -sialone and  $\text{Si}_3\text{N}_4$  in the filtration combustion mode, silicon of semiconductor purity is predominantly used. In TSC SB RAS it was proposed to use cheaper, available raw materials - ferroalloys - for the production of nitrides and sialon. In particular, to obtain  $\text{Si}_3\text{N}_4$  ferrosilicon (iron-silicon alloy). Nitriding of ferrosilicon is accompanied by heat release due to the reaction of interaction between silicon and nitrogen (756 kJ/mol). In [5] the results of the influence of aluminum additives (1-10%) on combustion patterns and phase composition of products during nitriding of a mixture of 40% ferrosilicon - 30% nitrided ferrosilicon - 30% zircon by SHS method are presented. It is shown that aluminum addition leads to the formation of  $\text{Si}_3\text{N}_4$ -based solid solution (SiAlON) in the combustion products. The aim of the work is to study the influence of aluminum additives on the combustion rate, nitrogen content, phase composition, morphology of synthesis products and properties of oxynitride composites obtained by nitriding of ferrosilicon-ilmenite mixture by SHS method.

To obtain oxynitride composites, nitriding of initial mixtures was carried out: 60% FeSi 2-Si + 25% FeSiN + 15%  $\text{FeTiO}_3 + x\text{Al}$ ,  $x = 1.0 - 10\%$ . Critical parameters at which the combustion process could not be realized were determined: nitrogen pressure less than 2 MPa, sample diameter less than 35 mm. It was found that the introduction of Al leads to a decrease or elimination of the  $\text{Si}_2\text{N}_2\text{O}$  phase in the combustion products as it increases in the initial mixtures. The phase composition of synthesis products at changing nitrogen pressure and sample diameter is represented by  $\beta$ - $\text{Si}_3\text{N}_4$ /  $\beta$ - $\text{Si}_3\text{Al}_3\text{O}_3\text{N}_5$ , TiN,  $\alpha$ -Fe. The chemical stage of interaction of ferrosilicon with ilmenite and aluminum additives in nitrogen atmosphere is shown. In the process of synthesis, aluminum-thermal reduction of  $\text{TiO}_2$  to Ti and subsequent nitriding of Ti to TiN are carried out. Aluminum promotes the formation of  $\text{Si}_3\text{N}_4$ -based solid solution (SiAlON) in the combustion products. The microstructure of the combustion products is represented by aggregates of small faceted crystals and formless formations. The open porosity (P, %) of the burned samples and the specific surface of powders (Sud.,  $\text{m}^2/\text{g}$ ) were determined depending on the amount of aluminum in the initial mixture. With increasing Al, the open porosity (61.9% - 52.7%) and specific surface area (0.88 - 0.52  $\text{m}^2/\text{g}$ ) decreased.

### REFERENCES

- [1] A. M. El-Amir Ahmed et al., "Sialon from synthesis to applications: an overview," Journal of Asian Ceramic Societies, no. 9, pp. 1390–1418, 2021, doi: 10.1080/21870764.2021.1987613.
- [2] N. V. Nikonorow et al., "Inorganic phosphors in lead-silicate glass for white leds," Journal of optics and spectroscopy, no. 3, vol. 121, pp. 379–383, 2016, doi: 10.1134/S0030400X16090162.
- [3] Y. Zhang et al., "The synthesis of single-phase  $\beta$ -Sialon porous ceramics using self-propagating high-temperature processing," Ceramics International, no. 3, vol. 48, pp. 4371–4375, 2022, doi: 10.1016/j.ceramint.2021.10.188.
- [4] Q. Wang et al., "SHS of Eu<sup>2+</sup>-doped  $\beta$ -SiAlON phosphors: impacts of N<sub>2</sub> pressure and Si particle size," Ceramics International, no. 5, vol. 43, pp. 4456–4461, 2017, doi: 10.1016/j.ceramint.2016.12.095.
- [5] O. G. Kryukova et al., "Combustion of ferrosilicon – zircon mixtures in nitrogen gas: impact of aluminum additives," International Journal Self-Propagating High-Temperature Synthesis, no. 6, vol. 52, pp. 402–404, 2021, doi: 10.1007/s11148-012-9450-1.