

## Study of the influence of alumina on the modification of corundum-carbon composites

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A comprehensive study of imported alumina “Salox ALO DN-23” was carried out using the methods of infrared spectroscopy, X-ray fluorescence analysis and electron microscopy, which made it possible to obtain a number of important results. It is shown that the studied alumina contains a significant amount of  $\beta$ -alumina, and the presence of calcium-containing impurities in it activates low-temperature reaction sintering of the composite material with the manifestation of an antishrinkage effect, a decrease in open porosity and pore size. Such features contribute to an increase in strength, reduce gas permeability and prevent carbon oxidation, increasing the efficiency of pyrolysis. Experimental verification of the prototypes of corundum-carbon composite using the studied alumina “Salox ALO DN-23” confirmed the predicted effects.

**Keywords** alumina, corundum-carbon composites, X-ray fluorescence analysis, electron microscopy

**Дослідження впливу глинозему для модифікації корундо-вуглецевих композитів.** С.М. Логвінков, О.М. Борисенко, А.А. Івашура, Г.М. Шабанова, В.М. Шумейко, А.М. Корогодська

Проведено комплексне дослідження глинозему Salox ALO DN-23 із застосуванням методів інфрачервоної спектроскопії, рентгенофлуоресцентного аналізу та електронної мікроскопії. Показано, що досліджуваний глинозем представлений у значній кількості  $\beta$ -глиноземом, а наявність у ньому кальційвмісних домішок активізує низькотемпературне реакційне спікання композиційного матеріалу з проявом протиусадкового ефекту, зменшення відкритої пористості та розміру пір. Такі особливості сприяють збільшенню міцності, зниженню газопроникності та запобігають окисненню вуглецю, підвищуючи ефективність піролізу. Експериментальна перевірка дослідних зразків корундовуглецевого композиту із застосуванням дослідженого глинозему Salox ALO DN-23 підтвердила спрогнозовані ефекти.

### 1. Introduction

Recent innovative solutions were obtained for the composite material technologies of various types and purposes due to the comprehensive studies of the physicochemical properties of each original component and a detailed study of the mechanisms of their interaction both under the influence of technological parameters dur-

ing the composite production and operational factors in various operating conditions. The competitiveness of composites is ensured by the search for technological parameters that lead to the combination of individual interaction mechanisms into a new more efficient mechanism of the structure-&-phase self-organization with the manifestation of super-additive or emer-

gent properties of the obtained material. The formation of one or more specified properties in the material of a higher level exceeding its value in comparison to the appropriate weighted average value of each component enables the use of the composite in more severe operating conditions and provides increased versatility.

Carbon available in the composition of various composites can serve both as a matrix and as a filler, depending on its content, the type of precursors, the type of reinforcement and other technological factors [1–4]. For the production of widely-used carbon-containing composites capable to operate at high temperatures, natural but not artificial flake graphite of various fineness [1, 5–7] is used as filler. Its introduction contributes to an increase in thermal conductivity, compressibility, and corrosion resistance of metal and slag melts, along with a decrease in the temperature coefficient of linear expansion, friction coefficients, melt wettability, and maintain a high oxidation onset temperature ( $> 500$  °C) compared to other forms of carbon. Modern composites are multicomponent, poly-disperse, and carbon can be introduced into them by various precursors, making it possible to optimize the performance of individual technological operations and ensuring the availability of carbon with a different degree of crystallographic order in the product [6, 8, 9]. At the same time, there is a tendency to ensure the environmental friendliness of the production process and the safety of products made of composite materials. In particular, various types of carbon blacks and pitches are replaced by specially manufactured products based on coking materials; as for the widely used phenol-formaldehyde resins (due to their high coke residue (55 – 65 wt.%)), only those are used in which the content of harmful impurities (free phenol and formaldehyde) is minimal (within 0.01 – 0.02 wt.%). Phenol-formaldehyde resins serve as a temporary binder in the charge composition intended for pressing the composite. The binder is hardened in the presence of various catalysts during low-temperature heat treatment, and it can coke at higher temperatures, providing a strong bond for all components of the composite. However, additional ingredients are also required for the charge composition, in particular graphite wetting agents, dispersants, plasticizers, as well as antioxidant additives to prevent carbon burnout and the formation of secondary binders due to the synthesis of new compounds (SiC, MgO,  $\text{Al}_2\text{O}_3$ ,  $\text{MgAl}_2\text{O}_4$ , etc.) in the compos-

ite [2, 3, 9]. Neoplasms fill the pore space, reduce gas permeability and provide additional strengthening of the composite. For dispersion-strengthened composites, nanosized powders, including those based on carbon in the form of whiskers, fibers, fullerenes and nanotubes have recently found a wide application in technological practice [2, 4, 7].

Corundum is used as a component of the composites taking into account the high temperature of its congruent melting (2042 °C), high hardness (9 units on the Mohs scale), thermodynamic stability at high-temperature contact with aggressive gaseous media (including ionized ones), abrasion resistance, etc. Corundum is used for the production of dense and porous composites for structural and functional purposes, in particular shut-off valves for pumping devices, wear-resistant products, special electrical insulators, high-temperature thermal insulation, cutting tools, filters and membranes, catalyst carriers, endoprostheses, etc. Corundum is a high-temperature modification of  $\alpha\text{-Al}_2\text{O}_3$ , but it can be introduced into the composition of the composite by various precursors, such as alumina ( $\gamma\text{-Al}_2\text{O}_3$ ), aluminum hydrates and organic salts, sintered and electromelted corundum, tabular alumina,  $\text{Al}_2\text{O}_3$  whiskers and fibers, corundum microspheres, etc. [3, 10]. Like the carbonaceous component of composites, corundum can form a matrix or serve as a dispersed filler. Products for high-temperature operating conditions are made with a corundum matrix phase, and the properties of the corundum-carbon composite are largely determined by the content of corundum, the presence of precursors and their type, the fractional composition of the charge, the type of reinforcement with carbon and corundum components.

The purpose of this research was to carry out comprehensive studies of one of the precursors of the corundum component and to establish its influence on the formation of the physical and mechanical properties of the corundum-carbon composite for high-temperature operating conditions.

## 2. Materials and methods

The matrix base of the composite is tabular alumina, grade T 60/64 (content (mass %):  $\text{Al}_2\text{O}_3$  – 99,6;  $\text{Na}_2\text{O} \leq 0,4$ ) manufactured by Almatic GmbH (Germany) [11, 12] in the amount of 95 wt. % and three fractions (mm) introduced into the charge: 2 – 1, 0.5 – 0 and less than 0.045.

Corundum precursors were introduced in an amount of 2 wt. % and are presented in the basic version of the charge composition with a mixture of alumina: reactive, grade RG 4000 and calcined, grade ST 800 (Almatic GmbH (Germany) [11, 12]), and in the developed composition these were completely replaced by alumina, grade “Salox ALO DN-23” manufactured by MAL Hungaricum Aluminum Production and Trade Limited Company by Shares (Hungary). The antioxidant supplement is represented by 2 wt. % powder mixture of Al and Si (Elkem AS, Norway), fraction 150 – 0  $\mu\text{m}$ . All other components were introduced in excess of 100 % in identical amounts:

- graphite is represented by fractions of 40 and 71 microns (Graphit Kropfmuh AG, Germany) in the amount of 3.5 %;

- a mixture of liquid phenol-formaldehyde resin (brand SP1001/2-1, LTD TD “Ukrainian resins”, Ukraine) with a powder cured resin of the same type and a catalyst (9 wt.% of the amount of liquid resin) - hexamethylenetetramine (urotropine) was 8 wt. %;

- silicon carbide (fr. 150 – 0  $\mu\text{m}$ ) produced by OJSC “Abraziv” (Zaporizhzhia, Ukraine) was taken in an amount of 2 wt. %;

- a binder additive based on partially structured carbon “Carbores P” (“FCA Castrop-Ralxel” Co, Germany) with a content of fractions: less than 0.4 mm – more than 90 %, less than 0.08 mm – more than 50%, was 1 wt. %;

- superplasticizer “Castament FW-10” company “BASF Construction Polymers GmbH” (Germany) was introduced in the amount of 0.5 wt. %;

- microsilica, brand “Elkem Microsilica Grade 917U” company “Cofermin Roshstoffe GmbH” (Germany) was 1 wt. % and it permitted the content of particles with the size exceeding 45 microns within 0.5 wt. %.

The uniform mixing of the charge components was provided by the stepwise mode of introduction into the laboratory mixer and sufficient mixing time. The specimens (mm: 100  $\times$  100  $\times$  30) were pressed using the method of the double-sided pressing in a metal mold at a specific pressure of 30 MPa. The heat treatment mode included low-temperature curing of phenol-formaldehyde resin in the specimen composition and subsequent pyrolysis for coking carbon-containing precursors. Composite specimens were cut with a diamond tool, and physical and mechanical properties were tested according to the methods adopted for refractory

materials in accordance with European norms and international standards (EN, ISO).

Alumina “Salox ALO DN-23” was studied using the methods of infrared spectroscopy and X-ray fluorescence analysis (spectrometer “Avatar” and “Expert 3L”, respectively). To determine the elemental composition and to study the morphological features of the analyzed alumina, a JSM-6390LV scanning electron microscope with an energy-dispersive spectrometer AZtech Energy (X-max50 detector) was used. Powder samples for the electron microscopy analysis were prepared on an aluminum substrate in the form of the alumina suspension in isopropyl alcohol, as well as by gluing alumina particles to hydrocarbon polymer scotch tape to exclude the influence of the aluminum substrate of previously studied suspension samples. In some cases, for better identification of the relief features of particles and their aggregates, a compositional contrast mode was used for the electron microscope, permitting the fixing of “knocked out” or secondary electrons.

### 3. Results and discussion

The IR-spectrum recorded in the infrared frequency range had a trivial form, i.e. a sloping, monotonically ascending curve with a sharp rise near the limiting frequency of 4000  $\text{cm}^{-1}$ . The spectrogram did not reveal any maxima characteristic of the stretching vibrations of the hydroxyl group (-OH), and indicated the absence of aluminum hydrates in the analyzed sample. The sharply ascending branch at about 4000  $\text{cm}^{-1}$  is attributed to the vibrations of the Al-O bond, characteristic of only  $\text{Al}_2\text{O}_3$ . These conclusions are consistent with the data of the certificate issued by the supplier of alumina for the content of oxides (wt.%): not less than 99.5  $\text{Al}_2\text{O}_3$  and not more for the following oxides:  $\text{SiO}_2$  – 0.04;  $\text{Fe}_2\text{O}_3$  – 0.04;  $\text{Na}_2\text{O}$  – 0.3;  $\text{CaO}$  – 0.03.

The X-ray fluorescence analysis was planned to primarily identify the presence and type of impurities, and not their quantitative content in the studied alumina sample. Therefore, the sensitivity of the device in terms of the content of the main component was initially underestimated in order to increase the possibility of identifying the impurities. As a matter of fact, the results were obtained with a significant deviation in the quantitative content of chemical elements:  $^{13}\text{Al}$  – 87.268 $\pm$ 0.192;  $^{14}\text{Si}$  – 7.201 $\pm$ 0.158;  $^{16}\text{S}$  – 4.785 $\pm$ 0.125;  $^{29}\text{Cu}$  – 0.237 $\pm$ 0.010;  $^{30}\text{Zn}$  – 0.509 $\pm$ 0.011. Direct recalculation of these re-

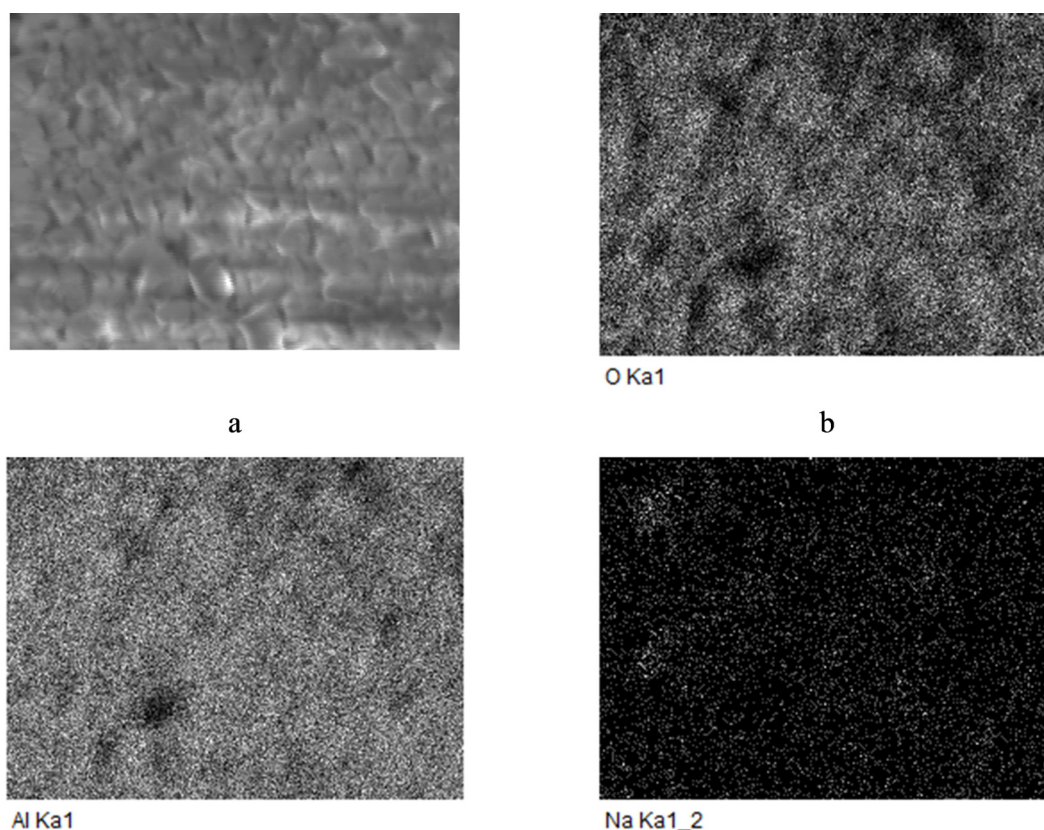


Fig. 1. Electronic image of a suspended alumina sample and the distribution maps of the main elements (O, Al, Na)

sults for the amount of the main component declared in the supplier's certificate (99.5 wt. %) makes it possible to obtain a more correct content of identified impurities (wt. %):  $^{14}\text{Si}$  – 0.283;  $^{16}\text{S}$  – 0.188;  $^{29}\text{Cu}$  – 0.09;  $^{30}\text{Zn}$  – 0.02. Taking into account the limited sensitivity of the device for the determination of light chemical elements (up to magnesium, in fact), there are no contradictions between these results (in the absence of  $^{11}\text{Na}$ ) among impurities and a slightly increased content of  $^{14}\text{Si}$  (when approximating it to the amount of  $\text{SiO}_2$  declared in the certificate). At the same time, the absence of  $^{30}\text{Ca}$  and  $^{26}\text{Fe}$  may be due to the locality of the studied sample and the significant limitation of their content, especially iron. Sulfur, copper and zinc identified in alumina can be both targeted microadditives and random impurities. Zinc in the composition of the studied alumina can act as a sintering activator due to the possibility of low-temperature interaction with  $\text{Al}_2\text{O}_3$  up to the formation of the garnet – zinc spinel  $\text{ZnAl}_2\text{O}_4$ . In addition, the reactive sintering proceeds with an increase in volume, and the compensation of shrinkage processes can be provided with a simultaneous decrease in microporosity. Sulfur can be considered as an accidental im-

purity formed due to the chemisorption of sulfur compounds from flue gases during the production of alumina. On the other hand, sulfur may be introduced into the alumina in the form of sulfates, for example sulfoaluminate, or the alumina is subjected to the acid washing. The availability of small amounts of sulfur in this case can have a positive effect on the temperature-time mode of alumina sintering. In addition, sulfur in the composition of sulfo groups of organic compounds is often added to dispersed powders as a special additive to improve grinding. It is difficult to identify a technologically significant role of copper in the composition of the studied alumina, and its admixture should be considered random.

During electron microscopic studies, the characteristics of alumina dispersion were taken into account according to the supplier's certificate: the specific surface of the powder is 3.0 – 4.5  $\text{m}^2/\text{g}$ , half of the powder particles have a diameter of up to 1.2 – 1.8  $\mu\text{m}$ , and 90 % of all powder particles do not exceed the diameter range of 3 to 5  $\mu\text{m}$ . Fig. 1a shows an electronic image of a suspended alumina sample selected for the microprobe analysis to define the content of the main chemical elements.

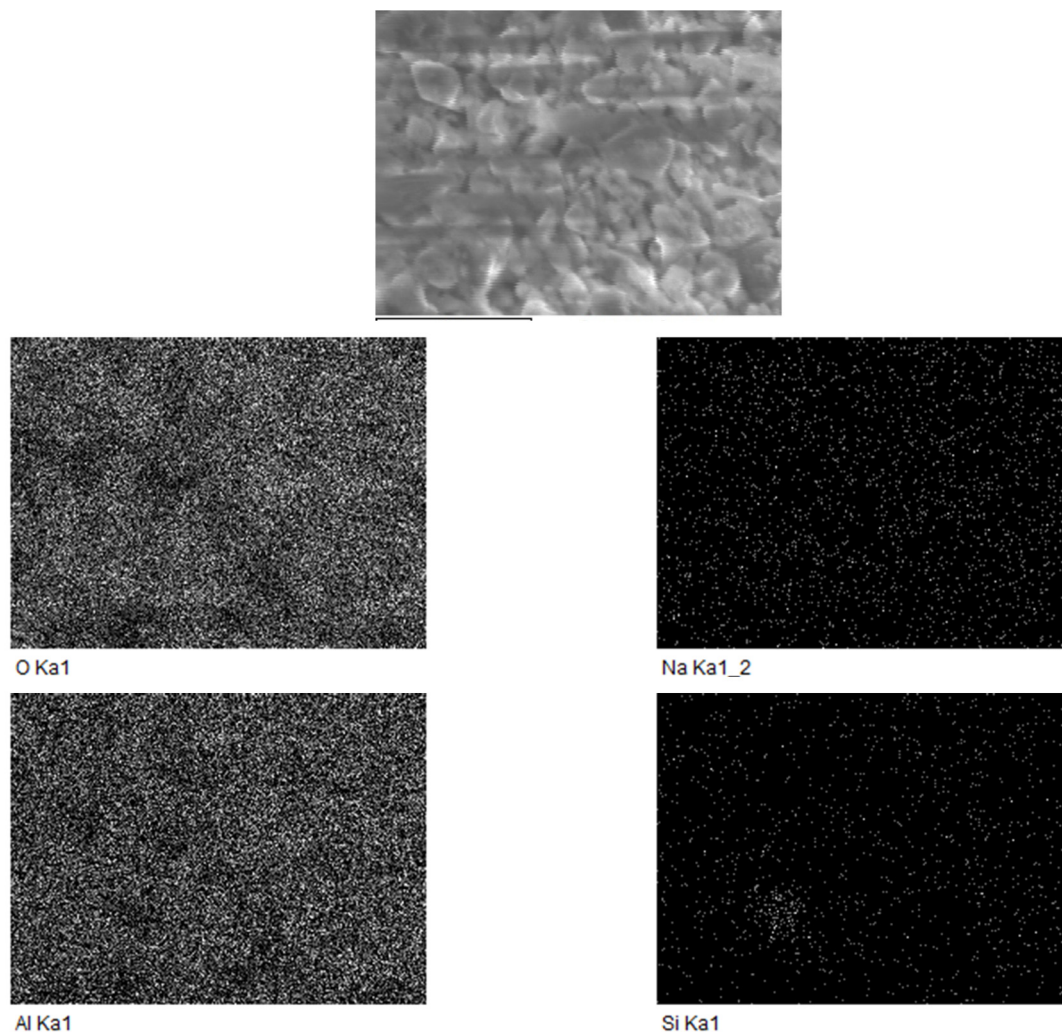


Fig. 2. Electronic image of a suspended alumina sample and the distribution maps of the main elements (O, Al, Na, Si)

Fig. Fig. 1 b – d show the distribution maps of oxygen, aluminum and sodium, respectively, as the main elements. The calculated concentrations of these elements according to distribution maps are as follows (wt.%): Al –  $52.29 \pm 0.11$ ; Na –  $0.89 \pm 0.15$ ; the rest is oxygen. In terms of oxides, the content in the sample (mass %):  $\text{Al}_2\text{O}_3$  – 98.79;  $\text{Na}_2\text{O}$  – 1.21. It is most likely that  $\beta$ -alumina ( $\text{Na}_2\text{O} \cdot 11\text{Al}_2\text{O}_3$ ) is present in a significant amount in the powder sample. This is evidenced by the relatively uniform distribution of sodium in the field of its map (Fig. 1d), where only in the upper left corner there is an area of increased distribution density. An additional microprobe analysis of the powder sample in this area made it possible to obtain confirming results of the elemental composition (in terms of oxides, wt %):  $\text{Al}_2\text{O}_3$  – 98.10;  $\text{Na}_2\text{O}$  – 1.90.

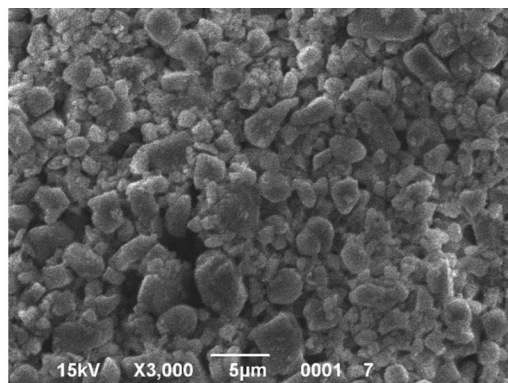
In another powder sample (Fig. 2), silicon is also fixed as the main chemical elements.

On the map of its distribution, an area of increased density is visible (to the left and below the center). In a similar section of the aluminum distribution map, the opposite situation is observed, which indicates the isolation of silicon from aluminum in this section; and the most probable phenomenon here is the individual presence of silica ( $\text{SiO}_2$ ) or elemental silicon but not the presence of aluminosilicate phases.

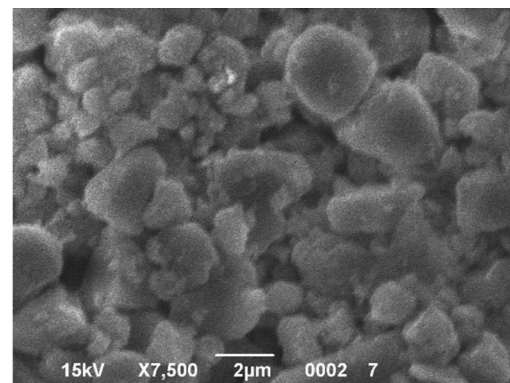
Micrographs of the powder sample on adhesive scotch tape (Fig. 3) confirm the fineness declared by the alumina supplier: the maximum particle size is up to  $5 \mu\text{m}$ , the predominant particle size is  $2\text{--}3 \mu\text{m}$ . Only individual particles have a polygonal shape, more often rounded particles are observed with the absence of

Table 1. Physical and mechanical properties of composite materials

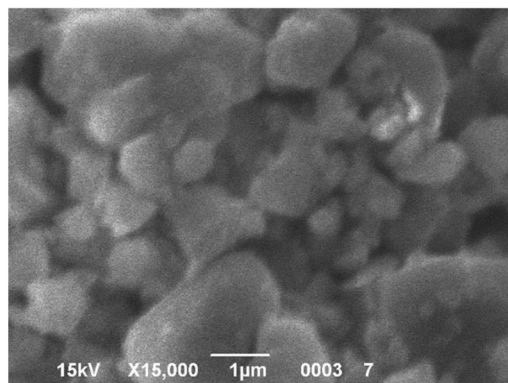
Properties	Base composite	Developed composite
After the low-temperature treatment		
Open porosity, %	3.9	6.0
Tensile strength at compression, MPa	117	129
Weight loss at calcinations, %	4.7	5.1
After the pyrolysis		
Open porosity, %	13.3	11.4
Tensile strength (MPa):		
- at compression	87	111
- in tension	18	21
Apparent density, g/cm <sup>3</sup>	2.97	3.01
An increase in open porosity, %	9.4	5.4
Decrease in tensile strength at compression, MPa	30	18



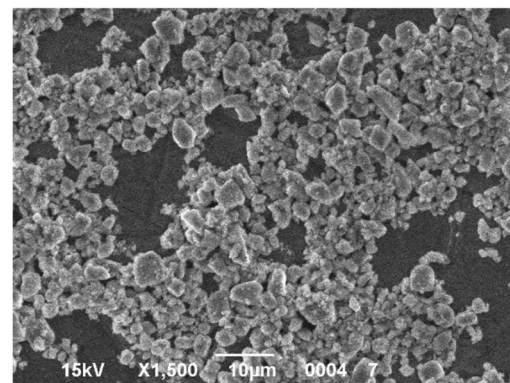
a



b



c



d

Fig. 3. Micrographs of the analyzed alumina in region 1

the elements of crystal faceting in the form of corners and edges.

The color of the particles is relatively uniformly gray; lighter rims are observed along the periphery, which are caused not by the presence of impurity phases, but by the misori-

entation of the electron beam at geometrically imperfect particle boundaries. Fig. 3 a – c show the noted morphological specific features of the particles of the powder sample with a successive increase in the resolution of the microscope for the central zone (Fig. 3a); and Fig. 3d shows

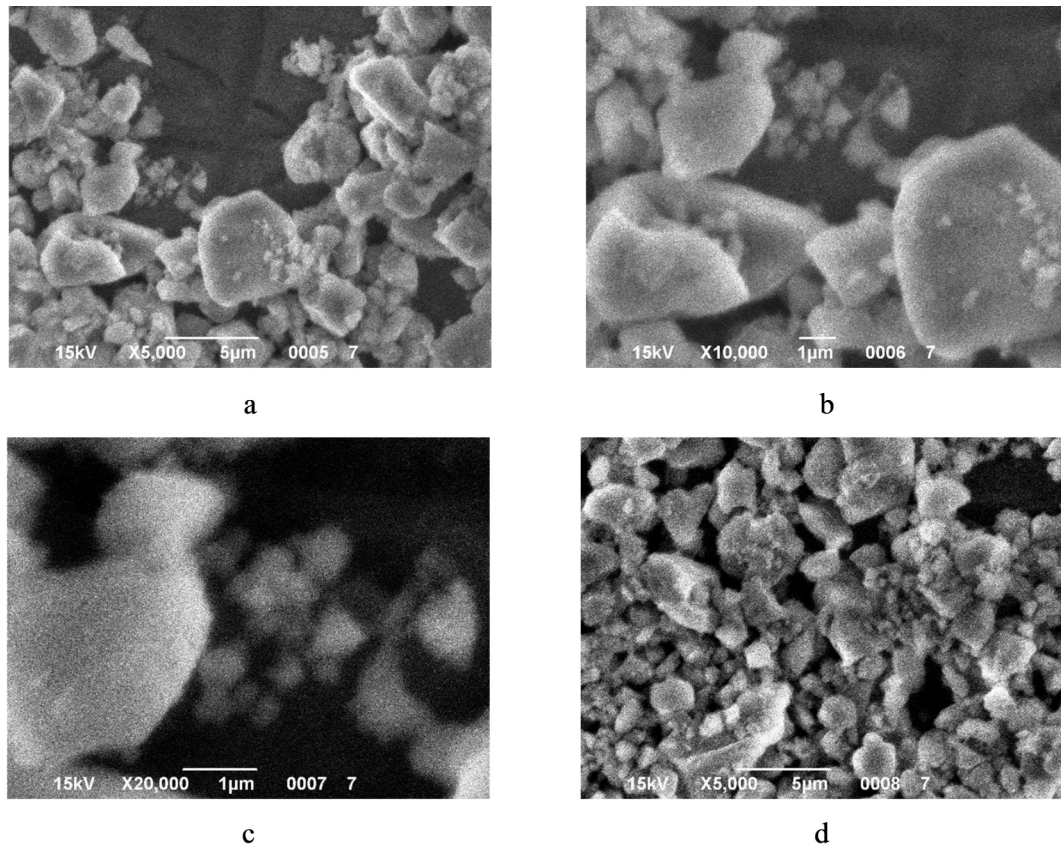


Fig. 4. Micrographs of the analyzed alumina in region 2

the most clearly seen light rims around the particles of the sample taken at a lower resolution. Only a bright spot (clearly visible in Fig. 3c, in the upper right corner) should be attributed to the impurity phase, and the light color of it is indicative of the presence of chemical elements in it with a higher atomic mass, in contrast to the main component.

In Fig. 4 a – c, with a successive increase in resolution, a large particle ( $\sim 5 \mu\text{m}$ ) with available relict angle ( $120^\circ$ ), preserved after the hexagonal faceting of a single crystal of corundum ( $\alpha\text{-Al}_2\text{O}_3$ ), as well as submicron particles in the powder sample are observed, and these are clearly distinguishable on the surface of the considered large particle (Fig. 4b) and to the left of it (Fig. 4c). In Fig. 4d, at a lower resolution, the polygonal nature of the morphology of the particles of the aluminum oxide powder sample is clearly visible. It should be noted that the large particle (Fig. 4a, b) has a tabular appearance; and on the outer basal plane an accumulation of brighter submicron particles is visible, which indicates that their composition is enriched in chemical elements with a higher mass number (probably CaO-containing phases). Such bright

particles form clusters in the edge zones of smaller alumina particles (for example, in the center of Fig. 4d).

This is an important circumstance, since it creates the prerequisites for reaction sintering with sequential synthesis and temperature increase during the pyrolysis of a number of calcium aluminates:  $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \rightarrow 12\text{CaO} \cdot 7\text{Al}_2\text{O}_3 \rightarrow 4\text{CaO} \cdot 3\text{Al}_2\text{O}_3$  (disproportioning at  $750^\circ\text{C}$  [13])  $\rightarrow \text{CaO} \cdot \text{Al}_2\text{O}_3 \rightarrow \text{CaO} \cdot 2\text{Al}_2\text{O}_3 \rightarrow \text{CaO} \cdot 6\text{Al}_2\text{O}_3$ . Moreover, the synthesis of  $\text{CaO} \cdot 6\text{Al}_2\text{O}_3$  occurs with an increase in volume, and it has a positive effect on compensating the shrinkage processes and minimizing open porosity with a sharp decrease in the pore size. It should also be taken into account that the presence of sulfates (sulfur was identified in the studied alumina by X-ray fluorescence analysis) enables the formation of calcium sulfoaluminate  $3(\text{CaO} \cdot \text{Al}_2\text{O}_3) \cdot \text{CaSO}_4$  or  $\text{Ca}_4\text{Al}_6\text{SO}_{16}$ , and its formation is accompanied by an even more significant increase in volume, by 13 % when synthesized out of  $\text{CaO} \cdot \text{Al}_2\text{O}_3$  and  $\text{CaSO}_4$  [13].

In addition, the reaction sintering is supplemented by the participation of  $\beta$ -alumina (previously it was considered to be a solid solution

of  $\text{Na}_2\text{O}$  in  $\text{Al}_2\text{O}_3$ , later as the  $\text{Na}_2\text{O} \cdot 11\text{Al}_2\text{O}_3$ , compound, now it is established that it belongs to the  $\text{Na}_2\text{O} \cdot 11\text{Al}_2\text{O}_3$  compound with an incongruent melting point of 2273 K [14].  $\beta$ -alumina is capable of reacting with calcium aluminates prior to the sequential formation of the following ternary oxide compounds:  $\text{Na}_2\text{O} \cdot 8\text{CaO} \cdot 3\text{Al}_2\text{O}_3$ ,  $2\text{Na}_2\text{O} \cdot 3\text{CaO} \cdot 5\text{Al}_2\text{O}_3$  and  $\text{Na}_2\text{O} \cdot 4\text{CaO} \cdot 10\text{Al}_2\text{O}_3$  in the high-alumina region of the  $\text{Na}_2\text{O} - \text{CaO} - \text{Al}_2\text{O}_3$  system. Such branched mechanisms of solid-phase interaction form the physicochemical basis for the reaction sintering of the corundum component of the composite in parallel with the pyrolysis of the carbonaceous component.

The above-mentioned specific features of the composition, dispersion and morphology of the studied alumina particles served as the basis for replacing the previously used alumina RG-4000 and CT-800 in the charge of the basic version of the corundum-carbon composite. Table 1 gives the comparison of the main physical and mechanical properties of the base and developed composite materials.

Comparison of the properties of the base and developed composites shows the effectiveness of replacing alumina RG-4000 and CT-800 with alumina "Salox ALO DN-23" due to the positive effect observed in the achieved values of open porosity and the strength indices of the developed composite.

#### 4. Conclusions

The comprehensive investigation of the imported alumina "Salox ALO DN-23" carried out using the methods of infrared spectroscopy, X-ray fluorescence analysis and electron microscopy enabled the generation of a number of important results. The technological feasibility of introducing zinc- and sulfur-containing impurities into alumina is substantiated from the standpoint of their participation in the processes of phase- &- structure formation of the corundum-carbon composite. It is shown that the studied alumina contains a significant amount by  $\beta$ -alumina and calcium-containing impurities which activate a low-temperature reaction sintering of the composite material with the manifestation of an antishrinkage effect, a decrease in open porosity and pore size. These features contribute to an increase in strength, reduce gas permeability and prevent carbon oxidation, increasing the efficiency of pyrolysis. Experimental verification of the prototypes of the corundum-carbon composite using the studied alumina "Salox ALO DN-23" confirmed the pre-

dicted effects. The developed composite differs from the previous one by lower open porosity, higher density, and improved strength properties. The developed composite is characterized by a significantly limited increase in open porosity and a decrease in tensile strength at the pyrolysis stage. The improved properties of the developed composite material make it possible to operate the products made of it under more severe conditions and increase the versatility of its use for new technological processes..

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