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Structure and phase composition of new ceramic material.

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ABSTRACT

We discuss the characteristics of microstructure and phase composition of sintered ceramics from powders obtained by chemical dispersion of technical aluminium and alloy AK7 in comparison with ceramics from a powder of industrially produced aluminum oxide obtained by Bayer method ($Al_2O_3 \cdot 3H_2O$). Based on the data of scanning electron microscopy and x-ray phase analysis it is concluded that during the sintering of powders, obtained by chemical dispersion of silumin containing a considerable amount of nephelite is realized the mechanism of the zonal sintering, providing formation of "bimodal porosity".
Keywords: aluminium oxide, corundum, scanning electron microscopy, chemical dispersion, x-ray diffraction, bimodal porosity.

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INTRODUCTION

Previously developed at Moscow State Industrial University (now Moscow Polytechnic University) method of chemical dispersion of aluminum alloys to obtain alloy powders of boehmite as the starting raw material in the production of alumina ceramics, special purpose allowed us to obtain a number of new materials, greatly surpassing the properties of traditional alumina ceramics. As the primary explanation of the observed increase in the level of office properties we made a specific impact on the structure and properties of ceramic phases formed during sintering due to the presence in powders of one or another alloying element from a dispersed fame. In this paper, we show the potential impact of nepheline on the formation of microstructure of ceramics produced by sintering powder alluminium alloy, dispersed in an aqueous solution of sodium hydroxide.

RESULTS AND DISCUSSION

The surfaces of the specimen fractures (after testing of samples for pendulum pile driver) were studied using electron scanning microscope Karl Zeiss EVO 50.

Notation: sample 1 was obtained from the hydroxide industrial production - grade analytical grade; sample 2 was obtained from hydroxide synthesized by the decomposition of aluminium with alkali; sample 3 was obtained from hydroxide synthesized by the decomposition of silumin by alkali.

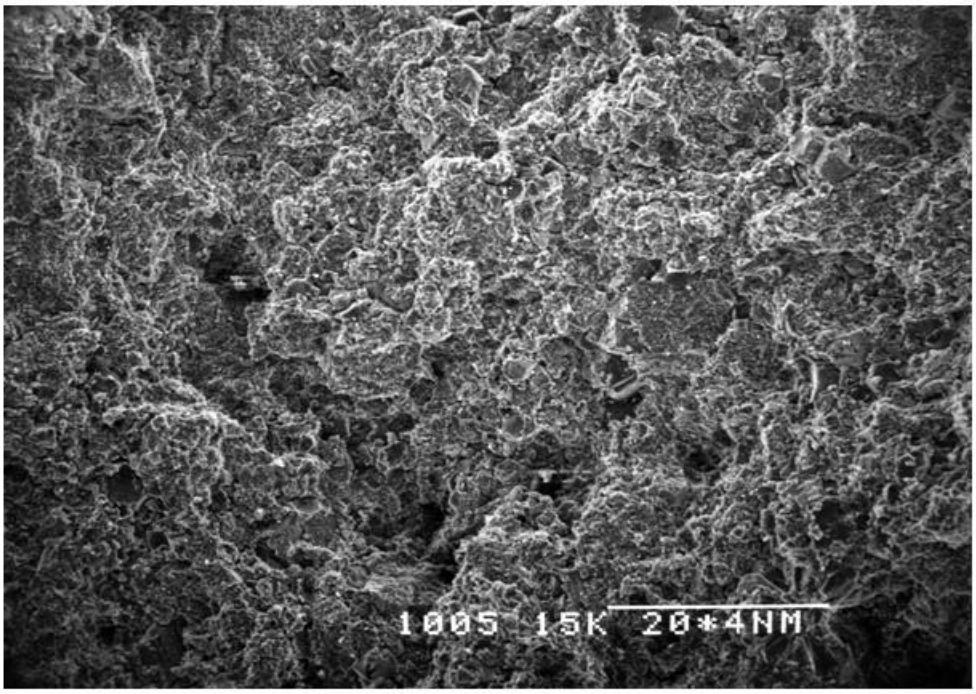

Fig. 1. Structure of fracture of a ceramic material obtained from industrial-grade aluminium hydroxide powder

Structure of fracture of sample 1 (Fig 1) is represented by porous grains (their sizes range from 20 to 100 microns). This is sintered in the amount of pressing of the original spherulites. They consist of micron and submicron particles and pores of the same size. There is a cut such grains associated with the desire of sintered powder system to lower the free surface energy. Other fragments - general view of fracture surface showing the presence of porous grains, intergranular pores, and cavities formed as a result of the tear of the grains upon impact.

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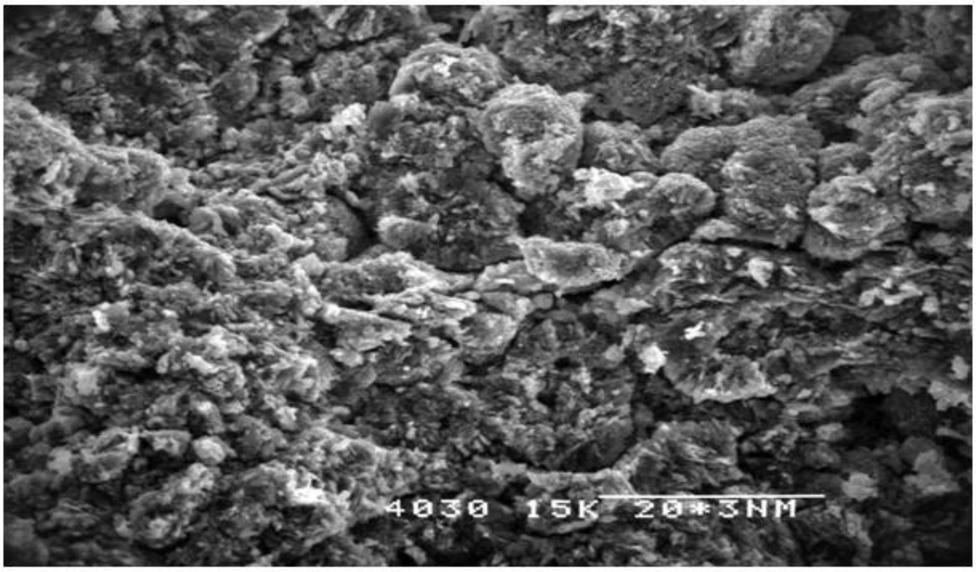

Fig. 2. Structure of the fracture of a ceramic material obtained from hydroxide synthesized by the decomposition of aluminum with alkali

The structure of the fracture surface of sample 2 (Fig. 2) is largely similar to the structure of sample 1. It is also made of porous grains consisting of submicron particles. The main difference (from sample 1) is significantly smaller grain size (1-10 microns). In addition, it is possible to notice the formation of gaps at the interface of these grains, which may be associated with the shock during the test and shrinkage of grains inside the volume in sintering (technological reason). Other fragments and the general appearance of the surface similar to the sample 1.

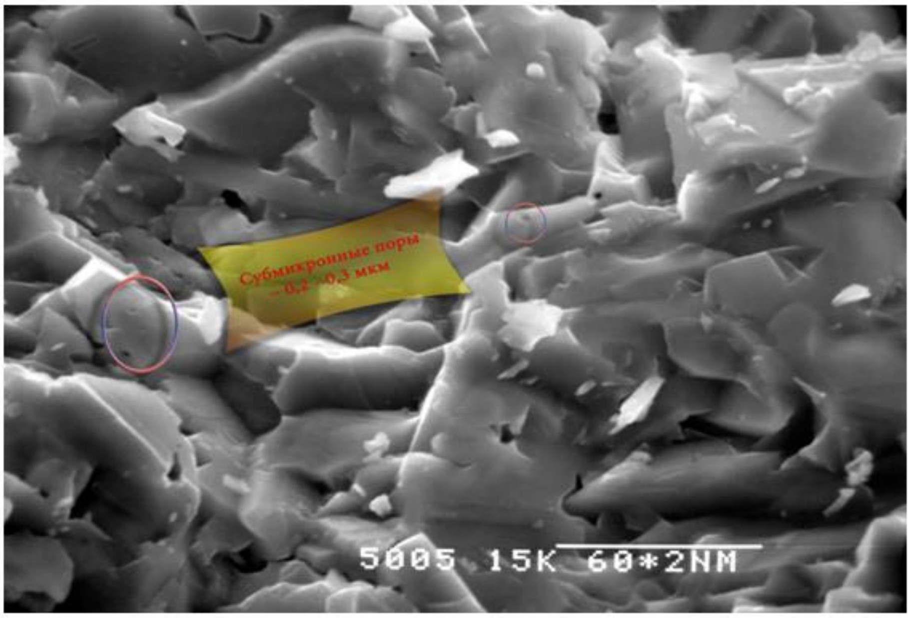

Fig. 3. Structure of a ceramic material obtained from hydroxide synthesized by the decomposition of silumin by alkali

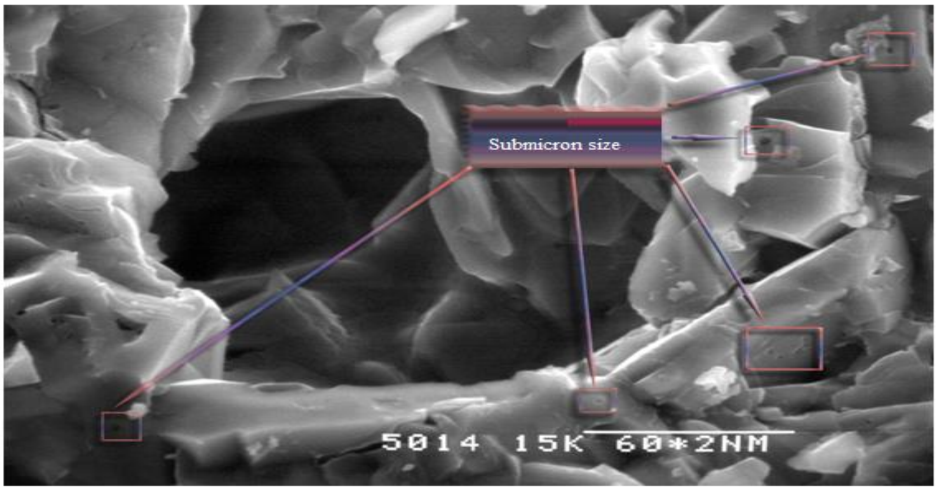

Fig. 4. Same as in figure 3, another piece

The structure of the fracture surface of sample 3 is presented with high-density platelet-shaped grains (1-10 μm) containing submicron pores. In this structure also fixed in the intergranular pores, the size of which is comparable with the grain size. Thus, this structure is characterized by a bimodal distribution of the pore dimensions (the so-called "bimodal porosity"). Leaf shape of the particles, in this case, due to the destruction of spherulites during grinding speck (after heat treatment of the hydroxide - 1350 g, 1 hour) to obtain a press powder. Preemptive destruction of the spherulites (compare samples 1 and 2) is probably connected with the action of internal stresses due to inhomogeneous phase composition (in speck, in addition to the main phase of aluminum oxide can be synthesized mullite $3Al_2O_3$ $2SiO_2$ и $2Al_2O_3$ SiO_2, main phase and mullite – are differ), it carried out x-ray analysis. The difference of the structure of the sample 3 (from the structures of samples 1 and 2) is associated with an extremely high activity to the sintering of powder obtained by the decomposition of silumin by alkali. The activation energy of sintering process of press from such a powder (to achieve the required density) is significantly lower compared to that of press of the other 2 powders.

Phase composition of sintered ceramics

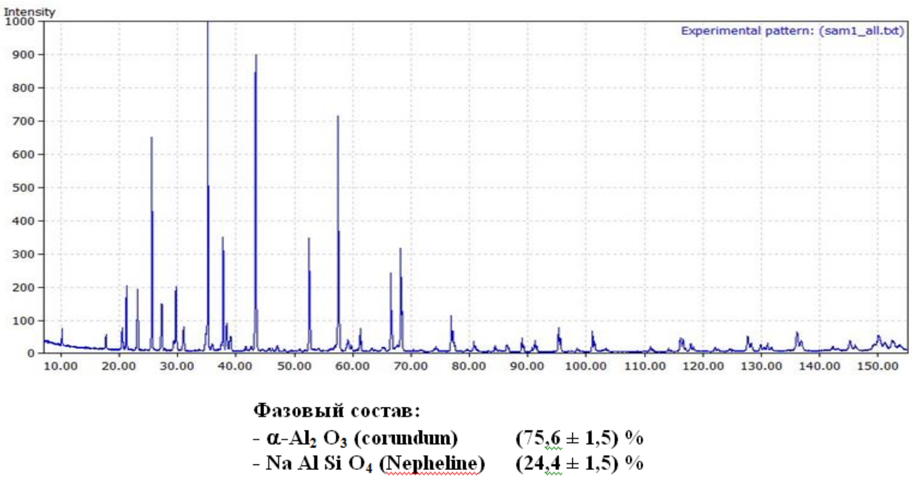

Fig. 4. Phase composition of a ceramic material obtained from hydroxide synthesized by the decompositionof silumin by alkali

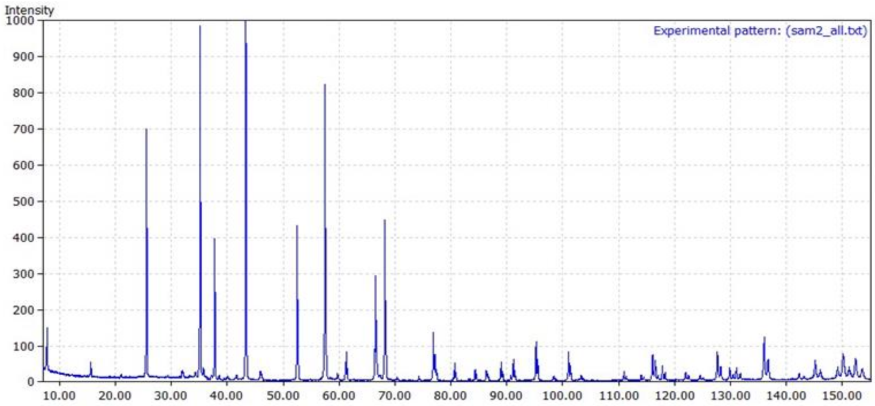

Fig. 5. Phase composition of a ceramic material obtained from hydroxide synthesized by the decomposition of aluminum with alkali

The formation of sodium aluminosilicate compounds (nepheline) in the composition of a sintered sample obtained from the hydroxide by the decomposition of silumin by alkali is interesting and new. Despite the fact that the main crystalline phase is alpha corundum, the role of the new phase can be very significant. The role of this phase is similar to the role of magnesium oxide in alumina industrial material - GLMC. It inhibits the growth of the main crystal phase alpha corundum as a result of collective recrystallization in sintering. This phase, according to the results, the uniformly distributed at the grain boundaries of alpha-alumina, resulting in lower surface energy of these boundaries.

I should say that taking as the object of dispersing silumin, we assumed that the possible effect of silicon on the structure and properties will be exactly in the formation of nephelite. It was supposed to synthesize nepheline (its T_{PL} = 1200ºC) to ensure zonal sintering with participation of a liquid phase. This liquid phase is able to "heal" submicron surface defects on the grains of corundum, and also help to suppress the growth of grains of corundum in the process of collective recrystallization.

Thus, the resulting material is a class of composites ceramic-on-ceramic - corundum in which the matrix phase. Type of bond in the composite between the matrix phase and nepheline is adhesive.

Based on the analysis of fracture surface of sample 3 and the X-ray fluorescence analysisdata it is possible to describe the following mechanism of the zonal sintering [2-4], providing formation of "bimodal porosity" (Fig. 6).

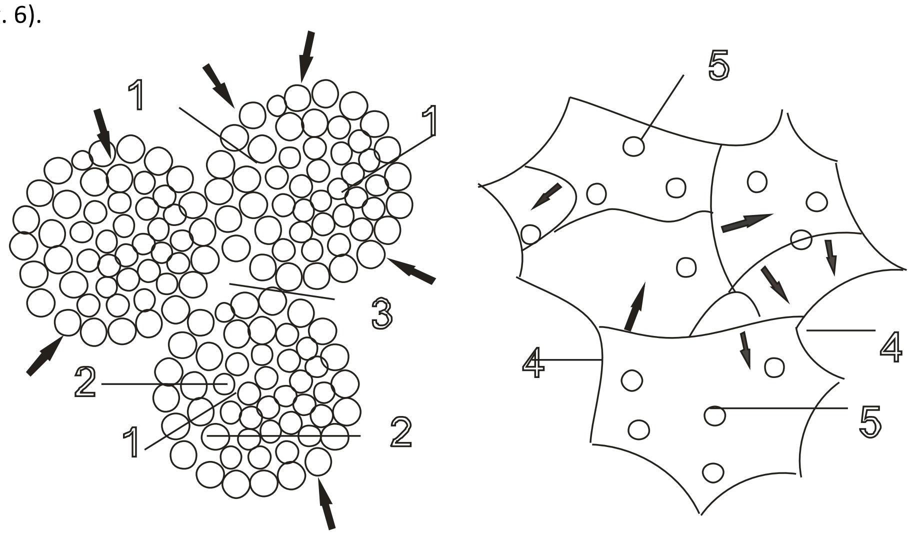

Fig. 6. Diagram of the zonal sintering

A. Structure of the compact before sintering	B. Structure of the sintered material
1. Nanoscale powder particles	4. Grain
2. Agglomerates of nanosized particles	5. Innergrains submicron pores
3. Interagglomerate pores	6. Grain closed pores
(Arrows indicate direction of compression of the agglomerates in the process of shrinkage during sintering)	(The arrows indicate the direction of migration of grain boundaries during collective recrystalize)

The nanoparticles of the initial powder form agglomerates (2) due to the action between the adhesion forces. In the sintering process, as the result of the shrinkage it occurs preferentially inside the compression volume of each agglomerate and the gap in the joints of the agglomerates with the formation of interagglomerate pores.

At the same time, due to the high specific surface area of the agglomerates and surface curvature of nanoparticles, a collective recrystallization is active. In the process flow of the agglomerates are transformed into grains (5) containing innergrains submicron pores (the presence of such pores is due to fluctuations in packing density of nanoparticles in the volume of agglomerates, resulting in a "gap" in the border regions of unequal density - similar to formation of interagglomerate pores).

Intergranular formation of innergrains closed pores in the sintered material due to the "collapse" of interagglomerate pores, with the movement of grain boundaries as a result of collective recrystallization. The proposed mechanism is consistent with the traditional theory of solid-phase sintering Frenkel-Pines.

CONCLUSION

It is established that chemical dispersion of aluminum alloys with silicon leads to alloy powders of ultrafine boehmite in the process of subsequent sintering auxiliary which forms the phase nepheline, which plays an important role in shaping the structure of ceramics. To describe the observable features of the formation of the microstructure of we proposed the mechanism of the zonal sintering, providing formation "bimodal porosity" of the ceramic.

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